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INVESTIGATION OF LITHIUM THIONYL CHLORIDE BATTERY  
SAFETY HAZARDS(U) GTE COMMUNICATIONS PRODUCTS CORP  
WALTHAM MA POWER SYSTEMS OPERATION R C McDONALD

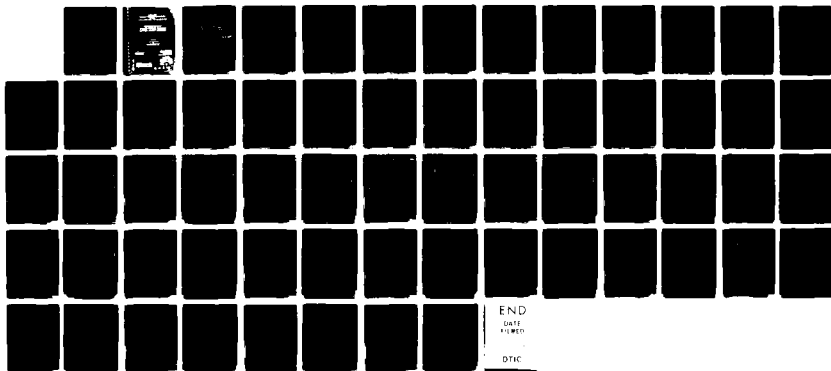
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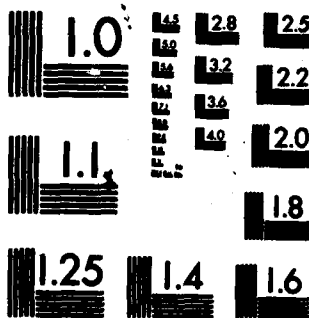
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**INVESTIGATION OF**  
**LITHIUM THIONYL CHLORIDE**  
**BATTERY SAFETY HAZARDS**



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Silver Spring, Maryland 20910

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## ABSTRACT

→ Glass  $\text{Li/SOCl}_2$  cells have been designed and built which will be used to discharge and overdischarge flat electrodes in sufficient but not excess electrolyte. The cells will be used for generation of electrolyte samples for chemical spectroscopy and overdischarged cathodes for morphological studies. A metal cell has been designed and built for overdischarge of flat electrodes.

E.S.R. Raman FTIR and UV/VIS fluorescence spectra have been taken of electrolyte at various stages of discharge and overdischarge. Two chemical species detectable with ESR are generated on discharge. The first resonance found also in electrolyte solutions saturated with sulfur, develops uniformly through discharge. The second as yet unidentified species becomes evident towards the second half of discharge increasing in concentration faster than sulfur. Following removal from the cell, the second species disappears as visible fluorescence increases in intensity the sulfur related peak remains.

Raman and FTIR spectra show a profusion of emission and absorption peaks respectively. Analysis of this data is still in process.

Preliminary optical studies of overdischarged cathode studies in cathode limited cells show that lithium dendrites do grow on the surface of carbon. ←

## I. INTRODUCTION

Reports of cell ruptures and explosions on overdischarge of lithium/thionyl chloride and lithium/sulfur dioxide batteries/cells have circulated for a number of years. Among the studies resulting from these events was one carried out by Abraham et al (1, 2) in which it was proposed that  $\text{Li}_2\text{S}$  is found in cathode limited cells and  $\text{SCl}_2$ ,  $\text{Cl}_2$  and a material with infrared absorption at  $1070\text{cm}^{-1}$  are formed in anode limited cells. The principal techniques employed were cyclic voltametric and infrared spectroscopy. Although explosions were found to occur in anode limited wound C cells on overdischarge, no specific chemical species or reaction mechanism was implicated.

A second study reported by Salmon et al (3) revealed the presence of a species, perhaps  $\text{Cl}_2\text{O}$ , which forms in  $\text{Li/SOCl}_2$  cells following removal of a relatively high applied overdischarge current to a cathode limited cell. It was speculated, still without a detailed mechanism, that decomposition of  $\text{Cl}_2\text{O}$  accounts for occasional explosions on cell reversal.

GTE Sylvania has performed a considerable number of overdischarge tests on D cells and 10,000 Ah Minuteman cells (5, 6, 8, 9, 10) in actual application series modes without observing cell hazards. However, these were  $1\text{ mA/cm}^2$  discharge application in anode limited cells. During such a controlled low rate overdischarge in anode limited cells, it is believed that the forced oxidation products (e.g.  $\text{SO}_2\text{Cl}_2$ ,  $\text{Cl}_2$ ) recombine rapidly with the forced reduction products (e.g. lithium metal dendrites) so that no sudden exothermic reaction takes place during or following overdischarge. However, this hypothesis must be verified experimentally. Furthermore, it has been known for some years that, as emphasized by Kilroy and James (4), intimate mixture of lithium, carbon and thionyl chloride are



extremely unstable. The implication is that in circumstances such as high rate overdischarge generate carbon/lithium matrices, a carbon catalyzed explosive reaction may occur between lithium and  $\text{SOCl}_2$  or other reactive species.

GTE has established a basis for understanding the  $\text{Li/SOCl}_2$  discharge mechanism (Schlaikjer) (7) and has performed preliminary studies on the infrared and Raman spectra of discharge products. The characterization of overdischarge products and secondary reactions would be a logical extension of this work.

This report summarizes the results achieved in the first quarter of Contract No. N60921-81-C-0229 "Investigation of Lithium/Thionyl Chloride Battery Safety Hazards". The principal objectives for this period were: 1) Design and build glass and metal test cells to generate liquid discharged and overdischarged electrolyte for spectroscopic analysis and for dendrite morphological studies; 2) Prepare some discharged and overdischarged electrolyte to test the feasibility of the major spectroscopic and handling techniques; 3) Design an in situ glass cell for collection of live spectra during discharge and overdischarge.

## II. CONSTRUCTION OF TEST CELLS

Two glass cells were built according to Figure 1. The cells allow for vacuum filling of electrolyte. The electrode stack consists of one double sided anode sandwiched between two carbon cathodes. Total electrode surface area is about  $30 \text{ cm}^2$  with the lithium and carbon thickness depending upon the particular experiment.

For initial studies at  $1 \text{ mA/cm}^2$  discharge rate, the electrode stock will be held between two hemicylindrical shims, so that the electrolyte volume required to immerse the electrodes will correspond to about 2 cc/Ah as is typical in production cells.

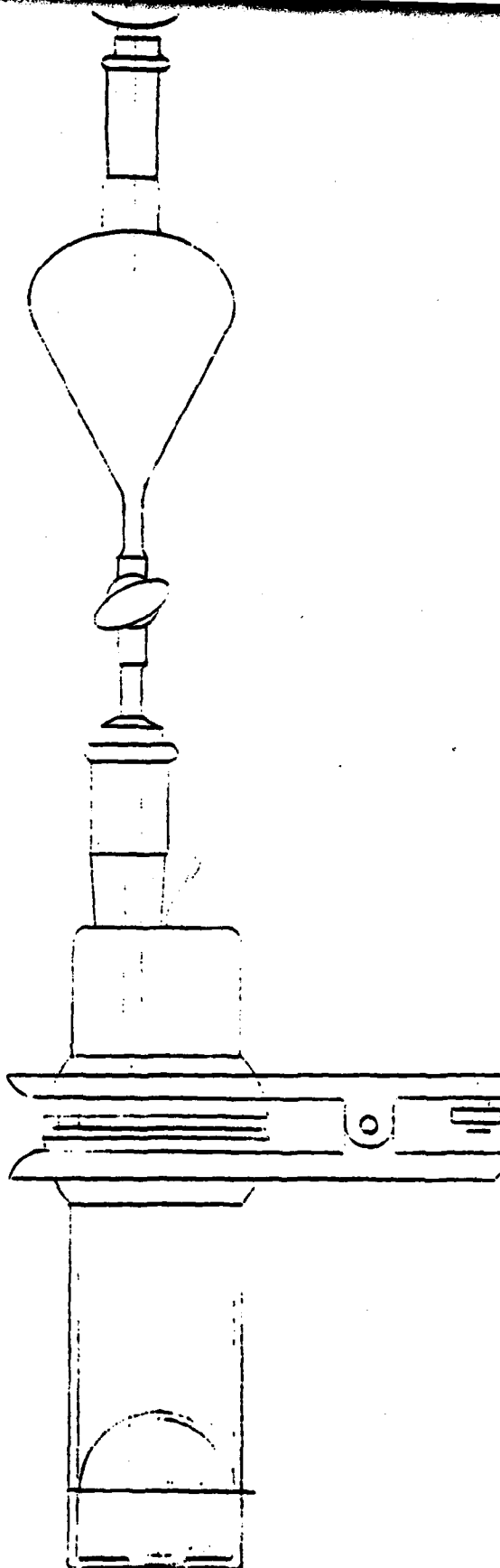
Tungsten G.T.M. feedthroughs with nickel tabs and substrates insure a hermetic seal and electrical continuity. A third feedthrough was provided to support a reference lithium electrode.

Constant current discharge and overdischarge will be performed in the glovebox and electrolyte samples drawn from the sidearm with a drawn glass capillary pipette.

A second cell was built identical to the first, except that a large flat glass window was pressed into the side so that morphological changes occurring on overdischarge can be microphotographed without disrupting the electrodes.

Figure 2 shows a cross-sectional view of the first heavy wall cell built for the more high rate and/or high temperature studies. The interior is sized for the same size stack and electrolyte volume used in the above glass cell. Three feedthroughs, one for a reference

FIGURE 1  
Glass Cell



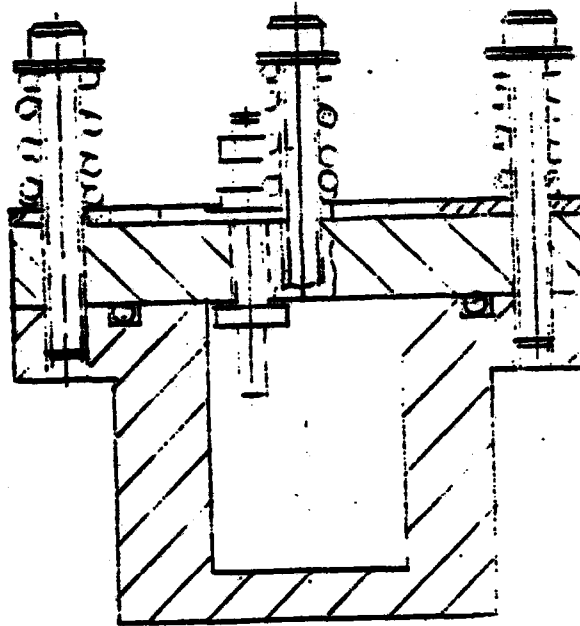


FIGURE 2

Metal Cell

electrode, pass through a halar flange. Pressure relief is provided by a set of matched springs and can be set by adjustment of the fixing screws.

A third type of cell, the in situ cell, is currently being prepared at the glass shop. Small electrodes with approximately  $.25 \text{ cm}^2$  surface area are mounted in pyrex. The body of the cell is quartz sealed to the pyrex feedthroughs with graded glass seals. The volume of excess electrolyte will be minimized although it is doubtful that the 2 cc/Ah ratio can be held while still adequately wetting the electrodes.

The design allows for the electrodes to be installed and for electrolyte activation in dry conditions. The completed cell will be fused shut. The first in situ cell will be sized for approximately 24 hours of discharge time at  $1 \text{ mA/cm}^2$ . This will allow for several Raman spectra to be collected over a two-day period without moving the sample or instrument settings. In a second cell, ESR spectra will be collected over the same period.

All solutions were kept below 20 ppm  $\text{H}_2\text{O}$ . Because the glass experimental cells were not ready for use, discharged electrolyte was obtained from D cells discharged at  $1 \text{ mA/cm}^2$  and room temperature. The cells were opened in the glovebox for electrolyte withdrawal. Overdischarged electrolyte was prepared by discharging and overdischarging demountable D cells (hereafter referred to as DT cells) which could be carefully opened in the glovebox and disassembled for examination.

### III. RESULTS

#### A. Electron Spin Resonance

E.S.R. spectra were collected on a Varian Model E-104A Band spectrometer with a modulation frequency of 10,000 Hz. Microwave power of 0.5 - 1.1 mW were used. Other instrument parameters are as follows:

Microwave Bridge	9.5 Ghz
Operating Frequency	8.8 - 9.6 Ghz
Frequency Calibration	$\pm$ 55 MHz
RF Power to Cavity	200 $\mu$ W maximum
Microwave range	40 dB (2 ms minimum)
AFC Lock Stability	1 part in $10^6$ at 0dB 1 part in $10^5$ at 40 dB
Line Ripple	1 ppm of set field or 3mG which ever is greater.

Spectra were routinely recorded between 3100 and 3500 Gauss at about 9.2 GHz which corresponds to a range of measurable electronic g factors of 2.120 to 1.878. Most primary ground state spin resonances fall in this range. In each case, a range of scale factors was used in order to search for weak signals.

All samples were taken in an Argon filled glovebox at 20 ppm H<sub>2</sub>O and sealed in 4 min high priority quartz tubes.

No resonances were detected for pure thionyl chloride, electrolyte (1.8M LiAlCl<sub>4</sub> in SOCl<sub>2</sub>), or electrolyte saturated with 1 atm. SO<sub>2</sub>. However, electrolyte saturated with sulfur gave a weak resonance at g= 1.9989.

No resonance were detected in fresh electrolyte. A strong resonance at g= 1.9983 develops early in discharge and a second at g=1.9896 toward the end of discharge. Upon cooling with liquid nitrogen, the first signal disappears, but reappears on warming. When the electrolyte containing two signals is removed from a cell and stored, the second signal disappears.

This coincides with the appearance of visible fluorescence.

Fifty percent overdischarged electrolyte from a cathode limited cell retains the above two signals in addition to a third at about  $g=1.9975$  overlapping the first signal. This third signal is accompanied by hyperfine splitting with  $\Delta H=86$  Gauss.

Copies of the E.S.R. spectra are included in the Appendix.

#### B. Ultraviolet/Visible Fluorescence Spectroscopy

To date, only discharged electrolyte has been examined with the fluorescence spectrometer. The instrument used is an Hitachi model with Xenon excitation. Both excitation and emission scanning modes are available with the instrument.

No fluorescence is observed from  $\text{SOCl}_2$ , electrolyte, or electrolyte saturated with either sulfur or sulfur dioxide. Electrolyte examined immediately following removal from a fully discharged cell (anode limited) shows no fluorescence. However, within 24 hours, this same sample, still sealed tightly in its cuvette begins to fluoresce. In fact, the solution slowly takes on a reddish cast.

Initially samples are excited at 514.5 m $\mu$  and fluoresce strongly from 560 - 660 m $\mu$ . This fluorescence remains and samples of discharged electrolyte from a year ago still show this effect.

#### C. Raman Emission Spectroscopy

Raman spectra were obtained on a Spex Spectrometer with a Spectra physics model 164 Ar<sup>+</sup> laser. The Spex Spectrometer consists of (1) a Spex Model 14 double monochromator, using two 1800 lines/mm gratings and scanning linearly in wavenumbers, (2) an EMI 9558 photomultiplier tube which was dry ice cooled to -60°C in a thermoelectric refrigerated chamber. D.C. ampli-

TABLE I  
RAMAN SPECTRA

<u>SOC1<sub>2</sub></u>	<u>FRESH ELECTROLYTE</u> <u>(1.8m LiAlCl<sub>4</sub> in SOC1<sub>2</sub>)</u>	
198 S	196	S
286 M	204	Shoulder
345 S	288	M
441 W. br	299	Sh
	347	S
490 M. br	364	S
1232 VW	441	M
	465	Shoulder
	493	M
	500	Sh
<u>S (SAT.)</u>	<u>30% OVERDISCHARGED</u>	
224 S. Sharp	210	M
251 W		
276 VW		
295 W (288)doublet (276 W)	280	W Sharp *
307 VW(307)                      295 )	305	W
333 VW		
349 M	332	VW
372 W	358	M
443 M, asym.		
478 S. Sharp	475	W
	509	M
874 VW		
1243 VW	1159	W
	1229	VW broad
	1344	VW (?) triplet



Table 1 (continued)

<u>100% DISCHARGED</u>	<u>25% DISCHARGED</u>	<u>SO<sub>2</sub> SAT.</u>
209 M	206 M	199.2 W
	223 VW (?)	
280 VW. Shoulder	277 *VS. Sharp	275 * Sharp
304 W		
333 VW	288 ) doublet ( .W 303 )	289 W. br. 348 M
354 S, asym.	350 S	371 * M. Sharp
473 W. Shoulder	373 * VS. Sharp	444 VW. Shoulder
505 M	464 W. Shoulder	494 W
601 VW (?)	506 M	1147 * W. Sharp
1162 W	1157 W	1154 W. br
1223 VW. br	1235 M	1231 W. br
1336 VW. br		

S - Strong; M- Medium, W- Weak, Br. - Broad

fication was used in this work and the output was dropped across a 100 K $\Omega$  resistor into a PAR 128 lock-in amplifier. Signal enhancement was further achieved by (a) signal averaging from a model NS-570 digital signal averager, (b) phase sensitive detection using a PAR chopper ( $\sim$  500 Hz) as the reference signal source.

Samples were sealed in a 4 mm O.D. quartz capillary, which was situated at the center of a silver-coated bulb. The collection optics of the monochromator were situated 90° relative to the incident beam. The 457.9nm line was used for excitation. The output power of the laser was 100 mw by a spectra-physics model 265 exciter. Scanning was done by a microprocessed spex compudrive CD2. The spectral slit width was typically 6 cm<sup>-1</sup> and scanning range from 170 to 1500 cm<sup>-1</sup>.

Table I summarizes the Raman spector collected thus far. Considerable attention was given to optimizing data collection conditions. It was found that cooling the detector with dry ice/methanol and using an optical chopper with correction for dark current, using a lock-in amplifier, significantly improved signal to noise ratio. Spectra are scanned in two sections - 100 - 750 cm<sup>-1</sup> and 750 - 1400 cm<sup>-1</sup> with 1060 channels available from each on multichannel analyzer.

Although it is a bit early, one can hazard a few qualitative remarks on these Raman Spectra. The electrolyte salt rather than providing a unique spectrum appears to be shifting most of the SOCl<sub>2</sub> peaks, suggesting a strong LiAlCl<sub>4</sub> - SOCl<sub>2</sub> complex in solution. Sulfur exhibits emission peaks at 224 and 478 cm<sup>-1</sup> when saturated in electrolyte, which do not appear in discharged or overdischarged electrolyte. This may be due to a different molecular form for sulfur when electrochemically generated in Li/SOCl<sub>2</sub> cells. Peaks 1154-1162 and 1336 are most probably due to SO<sub>2</sub>. Some of the electrolyte salt associated peaks such as 206-209. 299-304

and  $500\text{-}506\text{ cm}^{-1}$  increase in their relative peak heights as one would expect with the solvent being consumed and total solution volume decreasing. Measurements of lithium and aluminum concentration in fully discharged GTE cells (10,000 AH) show that the double salt concentration approximately doubles during discharge with detectable amounts precipitating in the porous carbon cathode.

#### D. Infrared Analysis

Instrument specifications for the MX-1E Nicolet spectrometer are given in Table 2. The infrared spectrum of electrolytes taken from discharging Li/SOCl<sub>2</sub> cells was reported by GTE in the literature in 1979 (J. Electrochem Soc. 126, p. 513). This technique was used to follow the concentration of sulfur dioxide as a function of discharge depth during ambient temperature constant current discharge. At the time, sodium chloride infrared cells with 0.1 mm cavities were being used with a Perkin-Elmer model 621 infrared spectrophotometer.

While the technique exemplified the power of infrared spectroscopy as both a qualitative and a quantitative tool for the analysis of air, water, and heat sensitive electrolytes, limitations to the technique existed. Sodium chloride does not transmit below about  $400\text{ cm}^{-1}$ , its lowest useful frequency being about  $500\text{ cm}^{-1}$ . The Perkin-Elmer 621, while being the best available instrument in its day, had limited resolution, particularly at low frequencies where wide beam apertures were required for a sufficiently strong signal.

GTE Laboratories recently acquired a Nicolet Model 3600 E Fourier transform infrared spectrophotometer, which is capable of reaching frequencies as low as  $200\text{ cm}^{-1}$ , and has far greater resolution throughout

the spectrum. Quantitative measurements can also be made more accurately, because the absorbance, rather than being affected by the speed at which the instrument responds, is calculated by the associated microcomputer from interference patterns produced with a monochromatic light source (laser).

We have recently found that disposable silver chloride cells with pathlengths of about 0.1 mm, can successfully be sealed. Samples may be taken and the cells transported without harm to the contents. Spectra of electrolyte samples taken on the Perkin-Elmer Model 700 before and after total immersion in water at room temperature were identical. The new cells may therefore be used with corrosive electrolytes in the Nicolet spectrophotometer without danger of harm to the expensive unit. Silver chloride is itself transparent to about  $400\text{ cm}^{-1}$ , and useable to  $312\text{ cm}^{-1}$ , a range far longer than previously possible. The average pathlength of individual cells may be accurately calculated using the interference pattern obtained by taking a spectrum of each empty cell. These cells should therefore make possible the accurate determination of the concentrations of dissolved materials. However, when attempts were made to follow changes in the electrolyte with time by periodically taking spectra of the same cell, reactions between the silver chloride and the electrolyte were found to have taken place. The electrolyte remains unchanged during a period of at least several hours, but not during an overnight period.

At the present time, qualitative spectra exist for the following solutions, using 0.1 mm pathlength AgCl cells on the Nicolet spectrophotometer.

TABLE 2  
MX-1E\* F.T.I.R. SPECTROMETER  
S P E C I F I C A T I O N S

Wavelength Range	4400-200 $\text{cm}^{-1}$
Typical Measurement Time for Quality 2 $\text{cm}^{-1}$ Spectrum (32 scans)	2 mins.
Maximum Resolution	.5 $\text{cm}^{-1}$
Wavelength Reference	He-Ne Laser
Wavelength Accuracy	0.01 $\text{cm}^{-1}$ (constant)
Abscissa Expansion	Infinitely Variable
Abscissa Repeatability	0.01 $\text{cm}^{-1}$ (constant)
Ordinate Range	0-100%T 0-3A
Ordinate Accuracy	0.1%T
Ordinate Expansion	Variable to 1000X
100% Line Flatness	+/- 0.2%T
Stray Light	0.02%
Noise ( 1 min measurement) 0.1% @ 2000, 1% @ 4000 $\text{cm}^{-1}$ (10 min measurement) 0.03% @ 2000, 0.3% @ 4000 $\text{cm}^{-1}$ }	@ 2 $\text{cm}^{-1}$ res.
Spectral Storage	800 4 $\text{cm}^{-1}$ Spectra
Recorder	Digital
Built-in Diagnostics	Standard
Digital I/O	RS-232C Standard

\*Extended range with CsI beam splitter (as opposed to KBr)

1. Discharged electrolyte, after a three-week stand at ambient temperature.
2.  $\text{SOCl}_2$
3.  $\text{POCl}_3$
4. 1.8M  $\text{LiAlCl}_4$  in  $\text{SOCl}_2$
5. 4), saturated at 1 atm with  $\text{SO}_2$
6. 4), saturated at room temperature with sulfur.

These spectra contained absorptions which have not previously been observed. In this discussion, we summarize by noting the differences between each of the following spectra:

1.  $\text{SOCl}_2$  and Electrolyte: In  $\text{SOCl}_2$ , a sharp absorption occurs at  $342\text{ cm}^{-1}$ . In electrolyte, this peak is surrounded by broader absorptions. In electrolyte, two strong, sharp absorptions at 615 and 650.5 are present, which are totally absent in the solvent. The  $\text{SO}$  fundamental at  $1231\text{ cm}^{-1}$  contains a shoulder at 1291 in the electrolyte, but not in the solvent.
2. Fresh and 3-week old Discharged Electrolyte: Discharged electrolyte shows many of the same absorptions, except most are shifted. The absorptions at 615 and 650.5 were far weaker and shifted from 1225 to 1204. The shoulder at 1291 disappeared. Added shoulders appeared at 1156, 1126, and 1080. A strong absorption appeared at 1332.
3. Electrolyte and Electrolyte with  $\text{SO}_2$ : Absorptions at 615 and 650.5 disappeared altogether. Strong, sharp absorptions appeared at 1337.5 and 1155 (compare 2., above).
4. Electrolyte and Electrolyte with Sulfur: Absorptions at 615, 650.5, 720 and 1291 totally disappeared.

We had hoped that  $\text{POCl}_3$  could be used as a solvent in order to examine the region near the SO fundamental at  $1204\text{ cm}^{-1}$  in discharged electrolyte. However, the PO fundamental was at 1297, with another absorption at  $1184.7\text{ cm}^{-1}$ . The spectrum contained ten other absorption between 1270 and  $220\text{ cm}^{-1}$ , which may limit the usefulness of  $\text{POCl}_3$  as a co-solvent to study absorptions in discharged or overdischarged  $\text{SOCl}_2$  electrolyte.

E. Overdischarge of a Carbon Limited, Flooded Cell

A cell with a single flat cathode about  $2\text{ cm} \times 2\text{ cm}$ , was discharged at  $5\text{ mA/cm}^2$ , ambient temperature, with an excess of lithium placed on either side of the cathode, at a distance far enough such that each surface of the cathode could be observed. The electrodes were immersed in a large excess of electrolyte in a glass cell with a flat window. On driving the cell past the point where the carbon polarized, lithium dendrites were seen to form on the cathode frame. The surface of the carbon lightened in color, as though coated with a white salt. On open circuit, the carbon electrode assumed a potential of about 0.1 volt cathodic to the lithium electrodes. When the circuit was closed again, long lithium dendrites were seen to grow on the surface of the carbon. Efforts are being made to photograph the edges and the surface of the carbon through the flat window.

#### IV. DISCUSSION

The first quarter efforts were primarily focused on technique development and characterization of baseline and discharged electrolyte spectra. The introduction of traces of water or air during sample handling does not seem to contribute to any of the spectra except for Raman and FTIR near  $2800-3200\text{ cm}^{-1}$  where hydrolysis products strongly emit and absorb respectively. It is felt that certain chemical changes are taking place following the sampling as shown by ESR resonances which change with time. These changes take place over a day to a week in sealed capillaries or cuvettes. Thus, the chemical changes truly represent secondary reactions of intermediates formed on discharge.

Despite the hyperfine splitting pattern observed on overdischarged electrolyte, it is probably wise to consider the three ESR resonances, which appear on discharge, and overdischarge, as three distinct species: A) formed on discharge and in saturated solutions of sulfur with  $g=1.9983$  averaged over 7 readings; B) formed on discharge with  $g=1.9896$  averaged over 5 readings; C) formed on overdischarge with  $g=1.9975$  with one reading.

At present, there is no evidence that intermediate reactions take place to any great extent during the 4-hour sample and travel time used so far. Use of the in situ cell will help clarify this important point.

Sulfur monoxide is expected to be Raman and infrared active at about  $1136\text{ cm}^{-1}$  (11). No such peak is observed in either FTIR or Raman spectra of discharged or overdischarged electrolyte. The observed  $g$  factors do not correspond to the known factor of 2.0022 for groundstate ( $X^3\Sigma^-$ ) sulfur monoxide (12).

Sulfur oxychloride radical,  $\text{SOCl}^\bullet$ , may be associated with one of the ESR resonances although no other collaborative spectroscopic evidence is available at this time.



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APPENDIX

ELECTRON SPIN RESONANCE  
and  
FOURIER TRANSFORM INFRARED SPECTRA

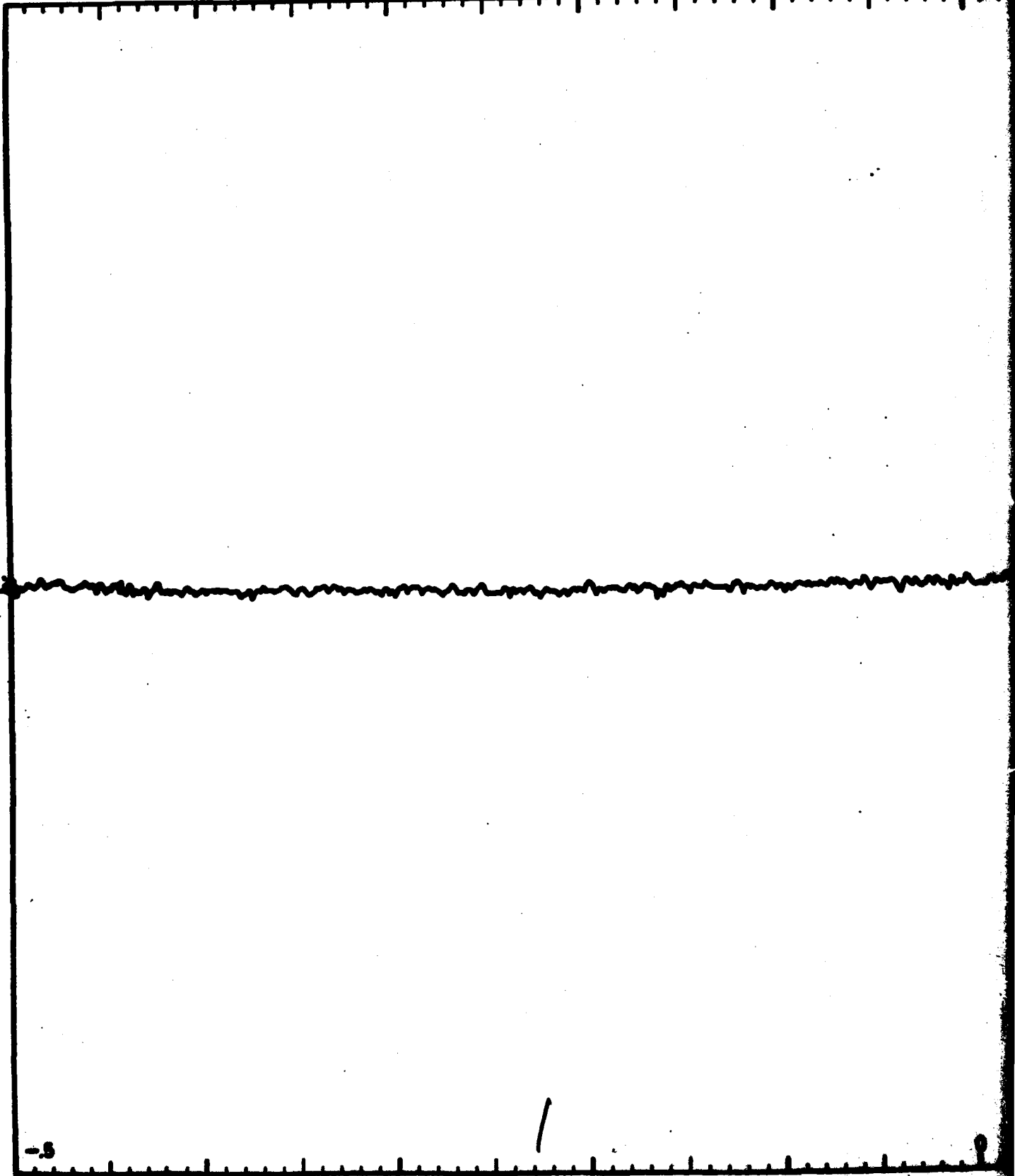
### ELECTRON SPIN RESONANCE SPECTRA

1. Saturated Sulfur in Electrolyte
2. Fresh 25% Discharged Electrolyte
3. Fresh 60% Discharged Electrolyte
4. Fresh 100% Discharged Electrolyte
5. Day Old 100% Discharged Electrolyte
6. 81 Day Old 100% Discharged Electrolyte
7. Fresh 50% Overdischarged Electrolyte (Cathode Limited)

### FOURIER TRANSFORM INFRARED SPECTRA

1. AgCl Cell Blank
2. Distilled  $\text{SOCl}_2$
3.  $\text{POCl}_3$
4. 1.8m  $\text{LiAlCl}_4$  in  $\text{SOCl}_2$  Electrolyte
5. Saturated Sulfur in Electrolyte
6. 1 Atm  $\text{SO}_2$  in Electrolyte
7. Fresh 25% Discharged Electrolyte
8. 3-day old 25% Discharged Electrolyte
9. Fresh 60% Discharged Electrolyte
10. Fresh 100% Discharged Electrolyte
11. 7-day old 100% Discharged Electrolyte

Scan Range  $4 \times 100$  g Time Constant  $0.25$  sec Modulation Amplitude  $2.5 \times 1$  g Receiver Gain  $6.3 \times 10^3$  Microwave Power  $42.5$  mW  
Field Set  $3300$  g Scan Time  $8$  min Modulation Frequency  $100$  K Hz Temperature  $RT$  °C Microwave Frequency  $9.287$  GHz



Operator J. Bennett

Date 12/30/81

Remarks

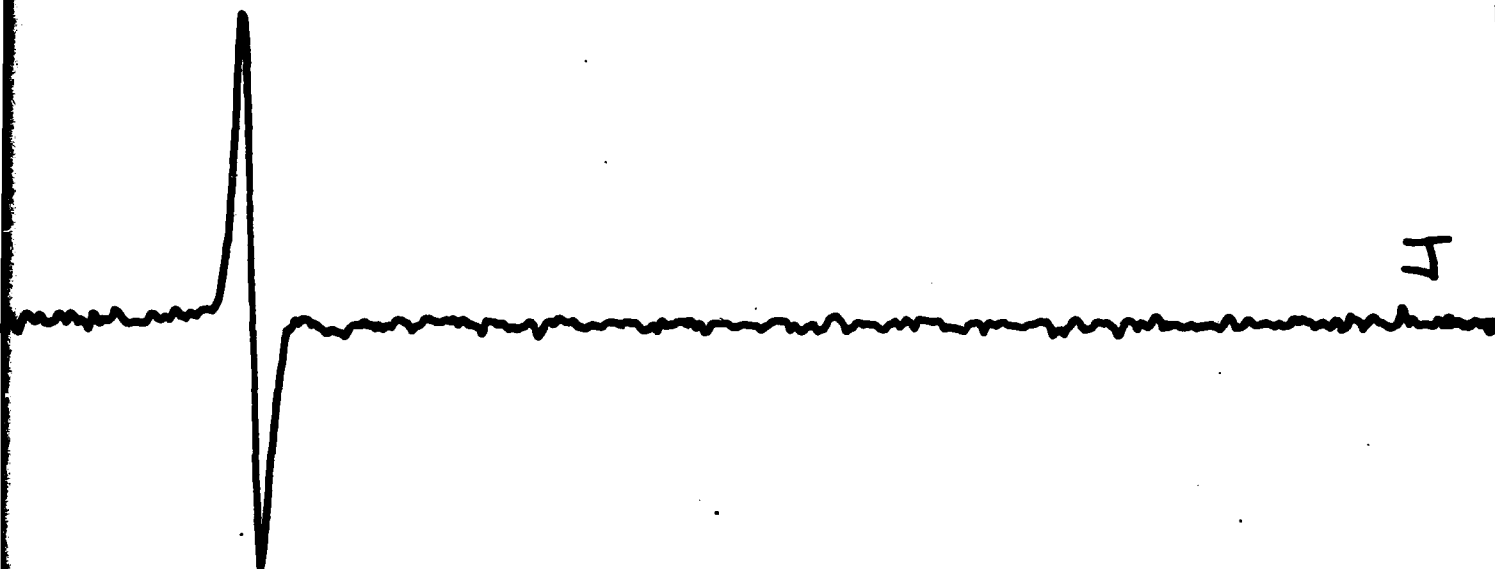
SR UNIT A

Sample

S in Electrolyte

Spectrum No.

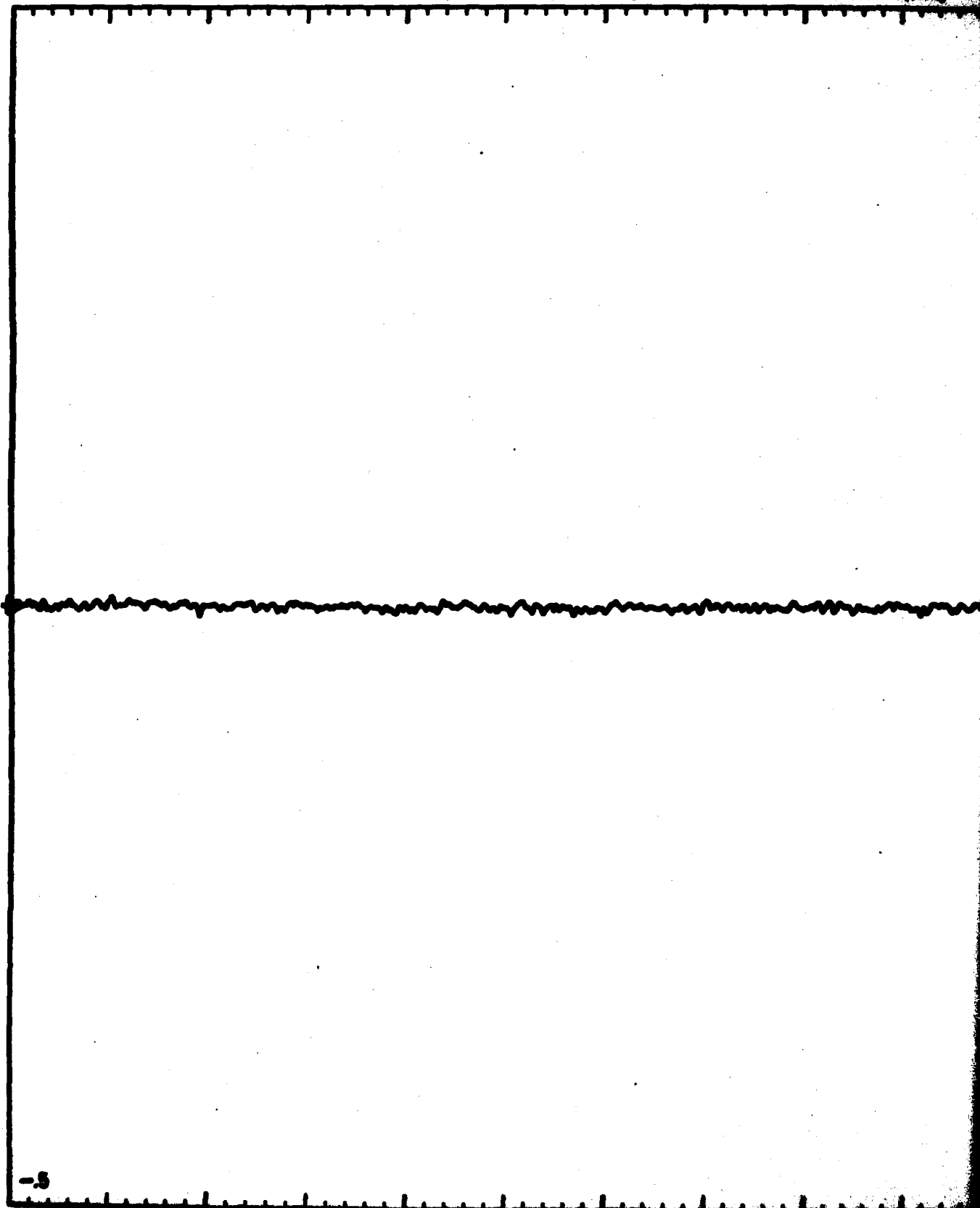
ESR-1  
+5



1

2

4 x 100 g 0.25 sec 2.5 x 1 g 6.3 x 10<sup>3</sup> 4.7  
Scan Range Time Constant Modulation Amplitude Receiver Gain Maximum Power  
3300 g hrs 8 min 100 K Hz RT °C 9.286  
Field Set Scan Time Modulation Frequency Temperature Measurement Frequency



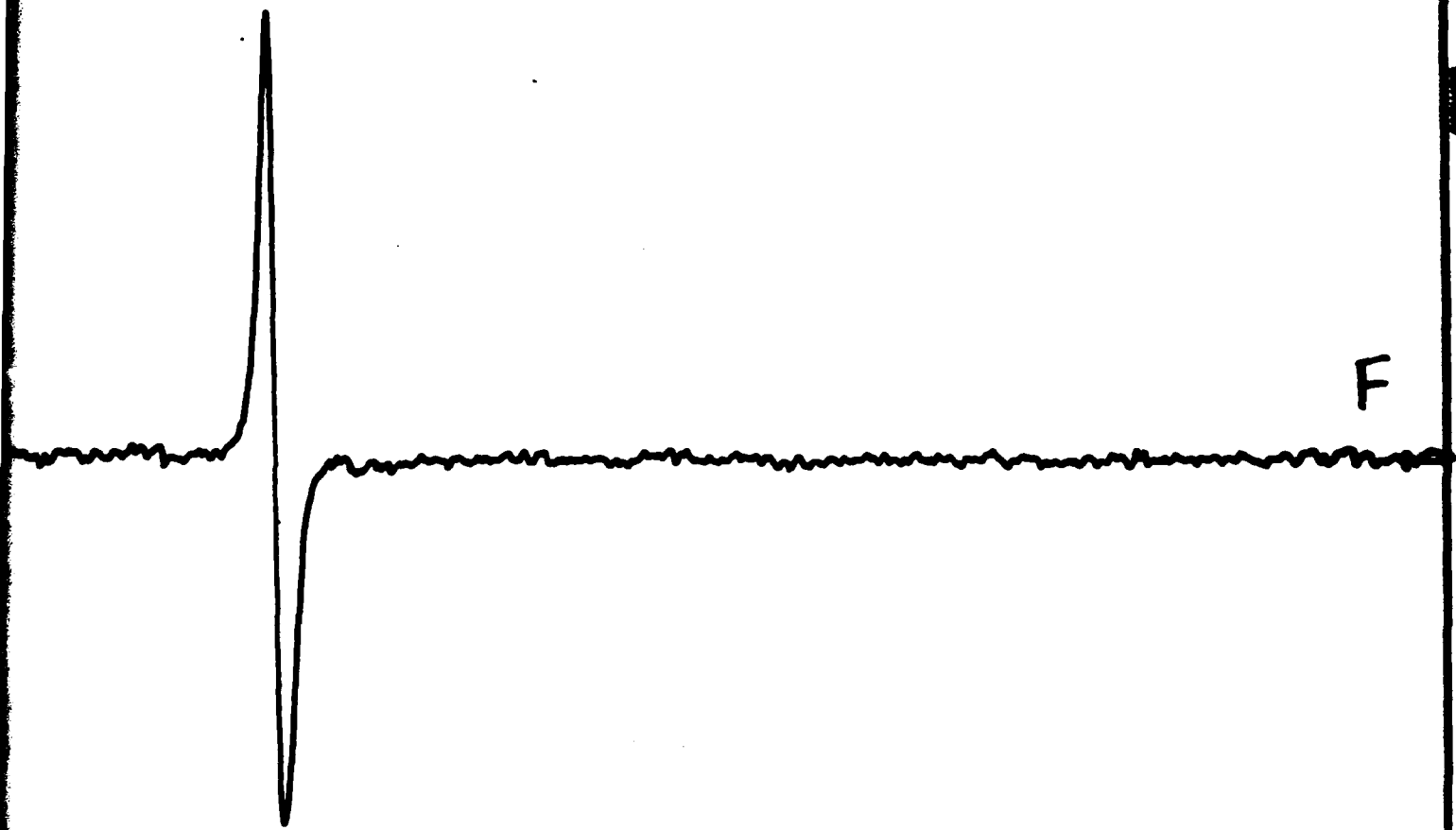
-5

Operator J. Bennett  
Date 12/30/81  
Remarks \_\_\_\_\_

SR QUART A

25% (12/18/81) Airbought Electra 76 0057

Spectrum No. \_\_\_\_\_



ESR-2  
+5

1 2



Scan Range  $4 \times 100$  g Time Constant  $0.25$  sec Modulation Amplitude  $2.5 \times 1$  g Receiver Gain  $6.3 \times 10^3$  Microwave Power  $4.7$  mW  
Field Set  $330c$  g Scan Time  $8$  hrs min Modulation Frequency  $100 K$  Hz Temperature  $RT$  °C Microwave Frequency  $9.284$  GHz



-5

J. Bennett  
Operator

12/30/81  
Date

Remarks

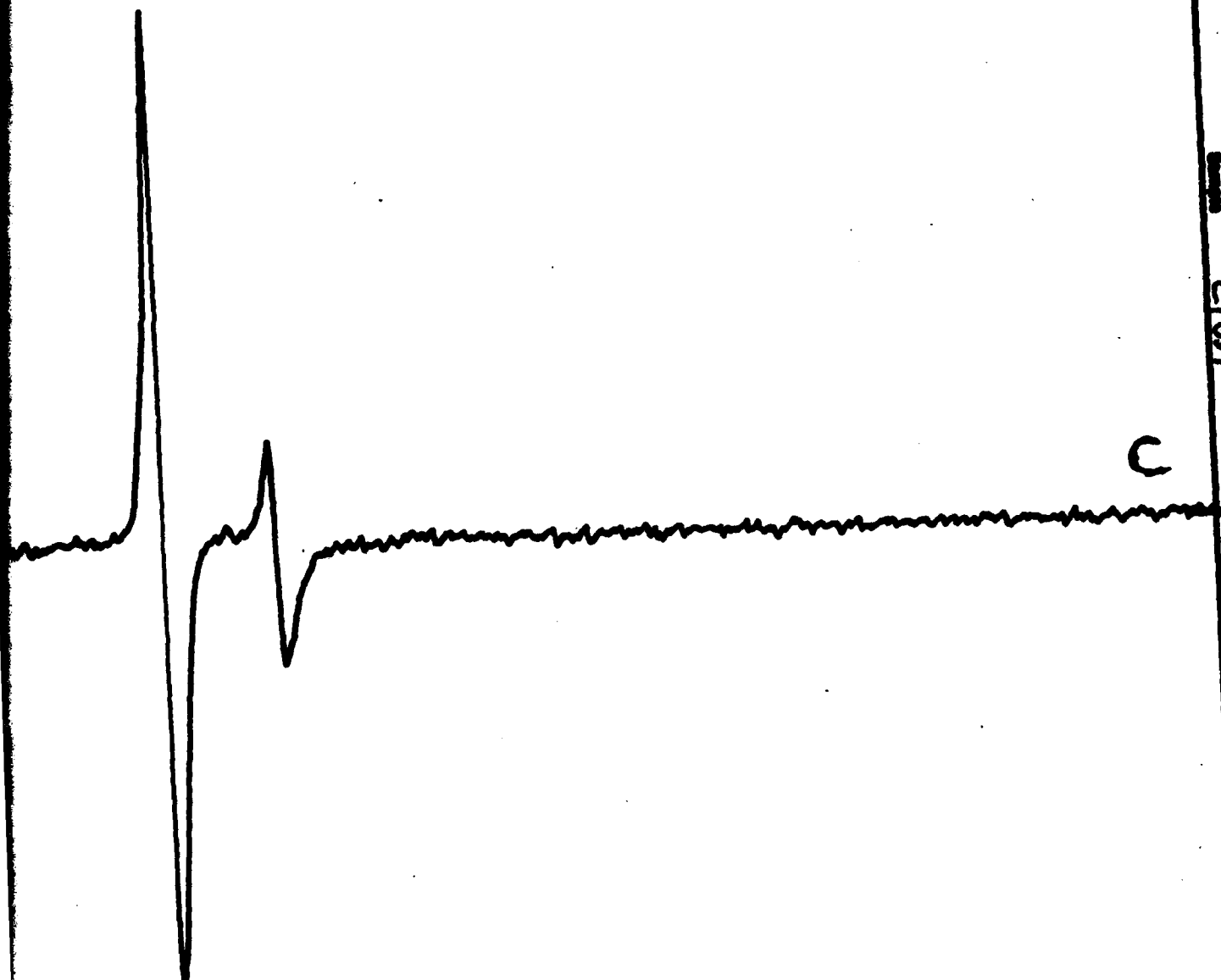
ESR CHART A

60% Dicumyl Peroxide (in hexyl) and Starch Peroxide  
Sample CTSI

Spectrum No.

ESR-3

+5



1

2

Scan Range 4  $\times 100$   $\mu$  Time Constant 0.25 sec Modulation Amplitude 2.5  $\times 1$   $\mu$  Receiver Gain 3.2  $\times 10^2$  Attenuator Factor 1.1 dB  
Field Set 3300  $\mu$  Scan Time hrs 8 min Modulation Frequency 100 K Hz Temperature AT  $^{\circ}$ C Attenuator Frequency 0 9.25  $\times 10^5$  kHz

MTW

Operator

*J. Bennett*

Q12

Date

*12/29/81*

Remarks

*D 2  $\frac{u}{m}$*

*E 3  $\frac{u}{m}$*

EPG Chart A

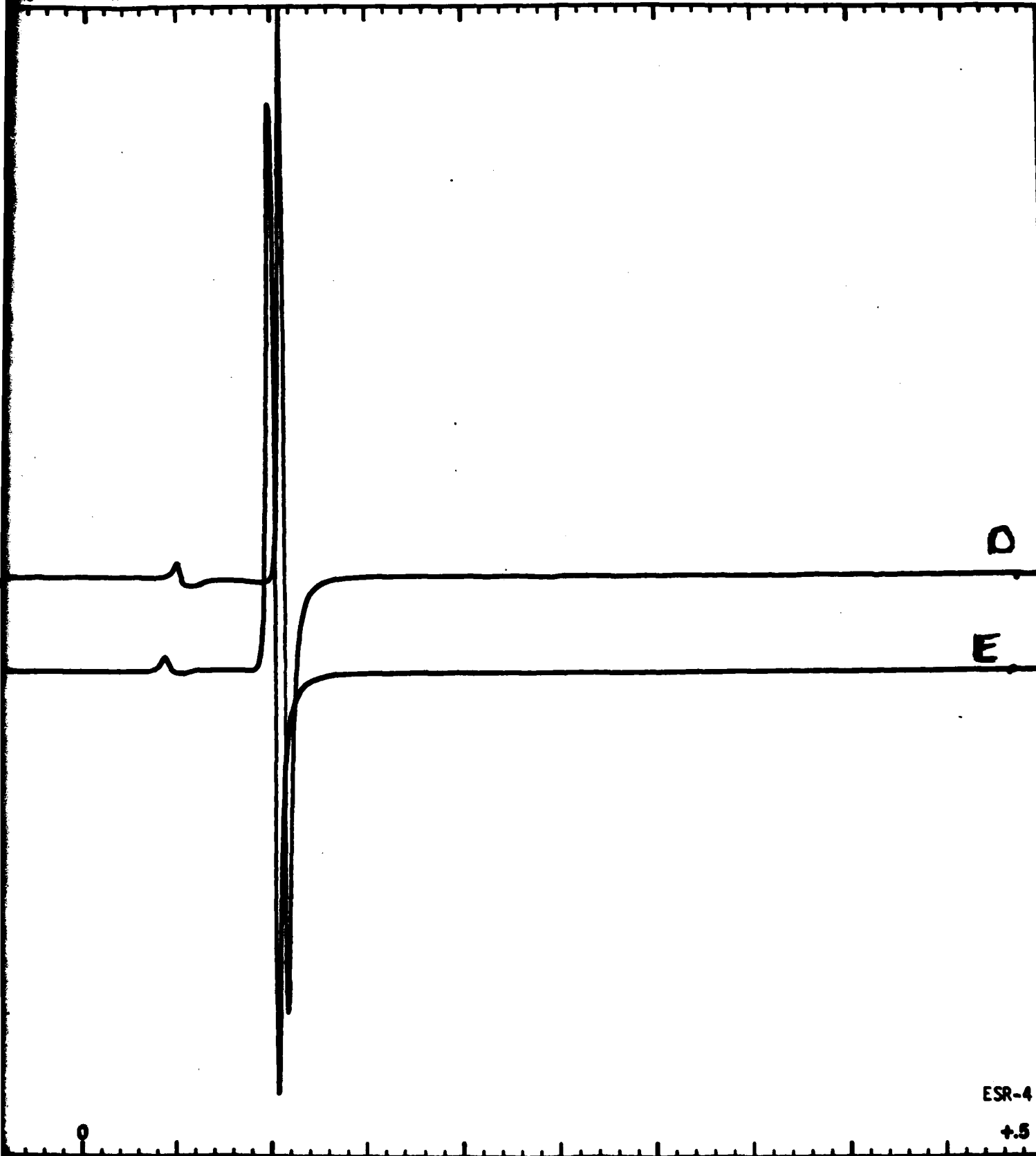
Sample

*100% Discharged Electrolyte + strong reference 12/29/81*

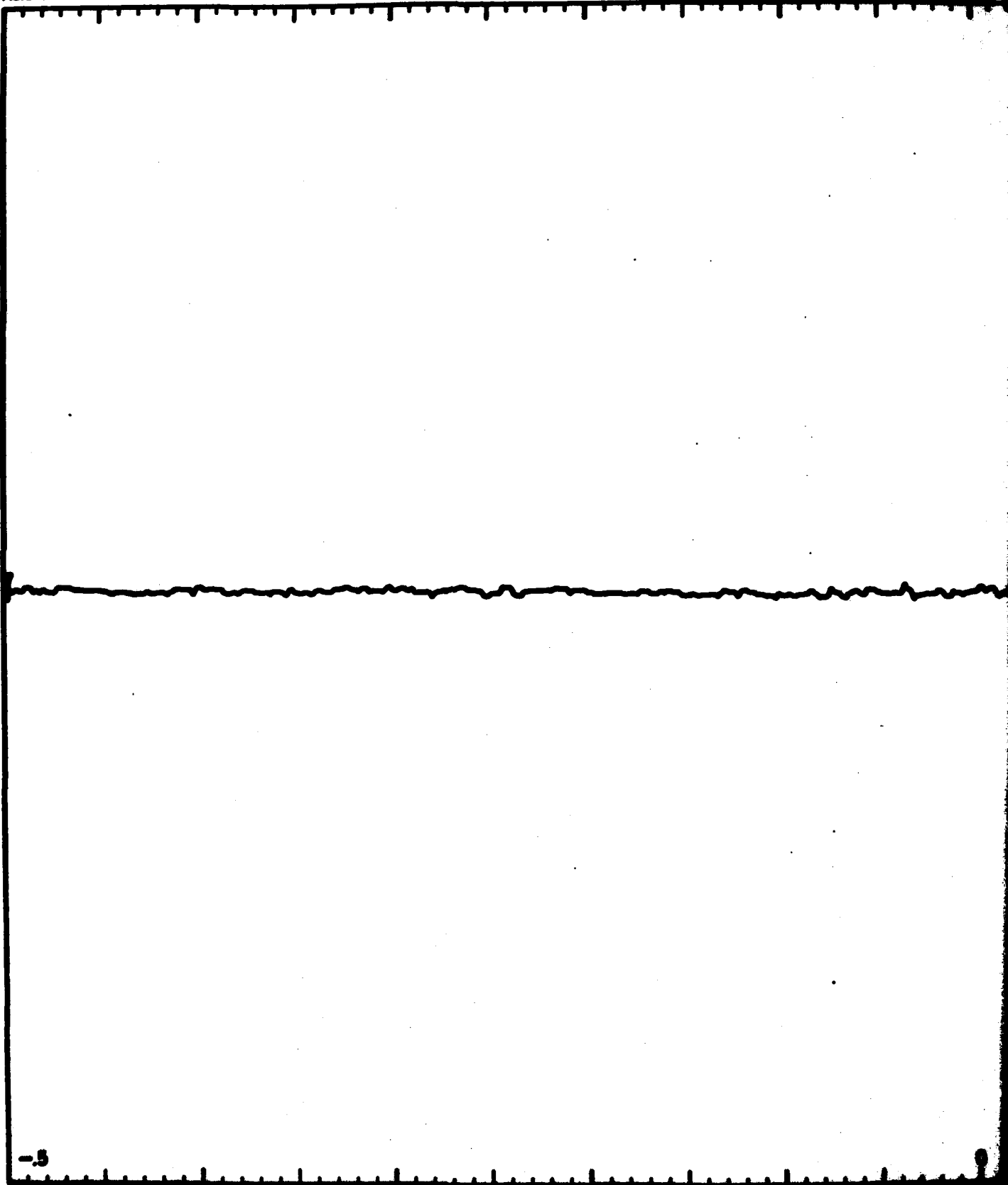
Spectrum No.

ESR-4

+5



4 x 100 g 0.25 sec 2.5 x 1 g 6.3 x 10<sup>3</sup> 0.92 dB  
Scan Range Time Constant Modulation Amplitude Receiver Gain Microphone Power  
3300 g hrs 8 min 100 K Hz RT 9.285 dB  
Hold Set Scan Time Modulation Frequency Temperature Microphone Frequency



J. Bennett  
Operator

12/20/81  
Date

Remarks

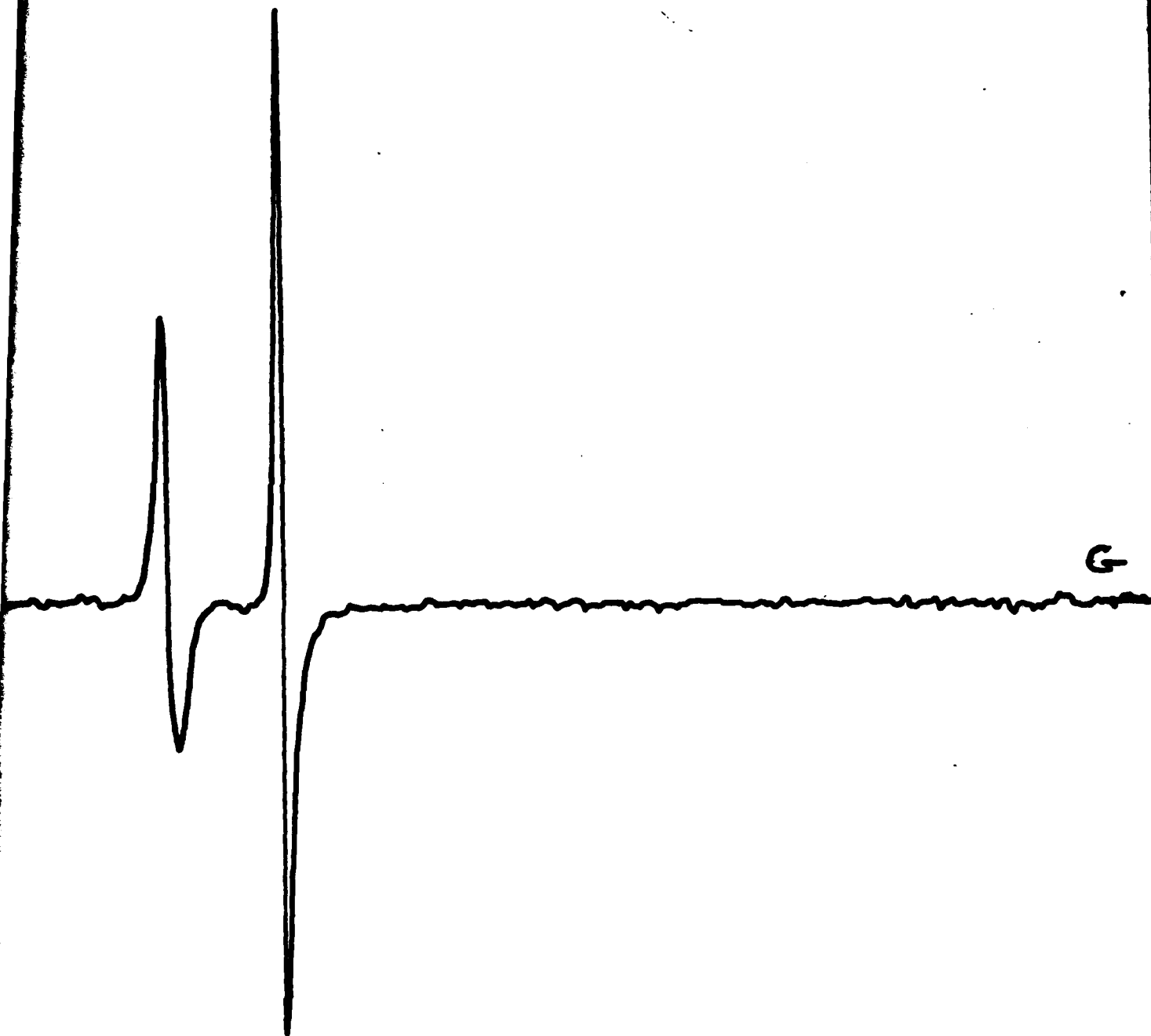
SPR QUART A

100% Discharged Electrolyte (n/a 9/81)  
Sample CT OSI

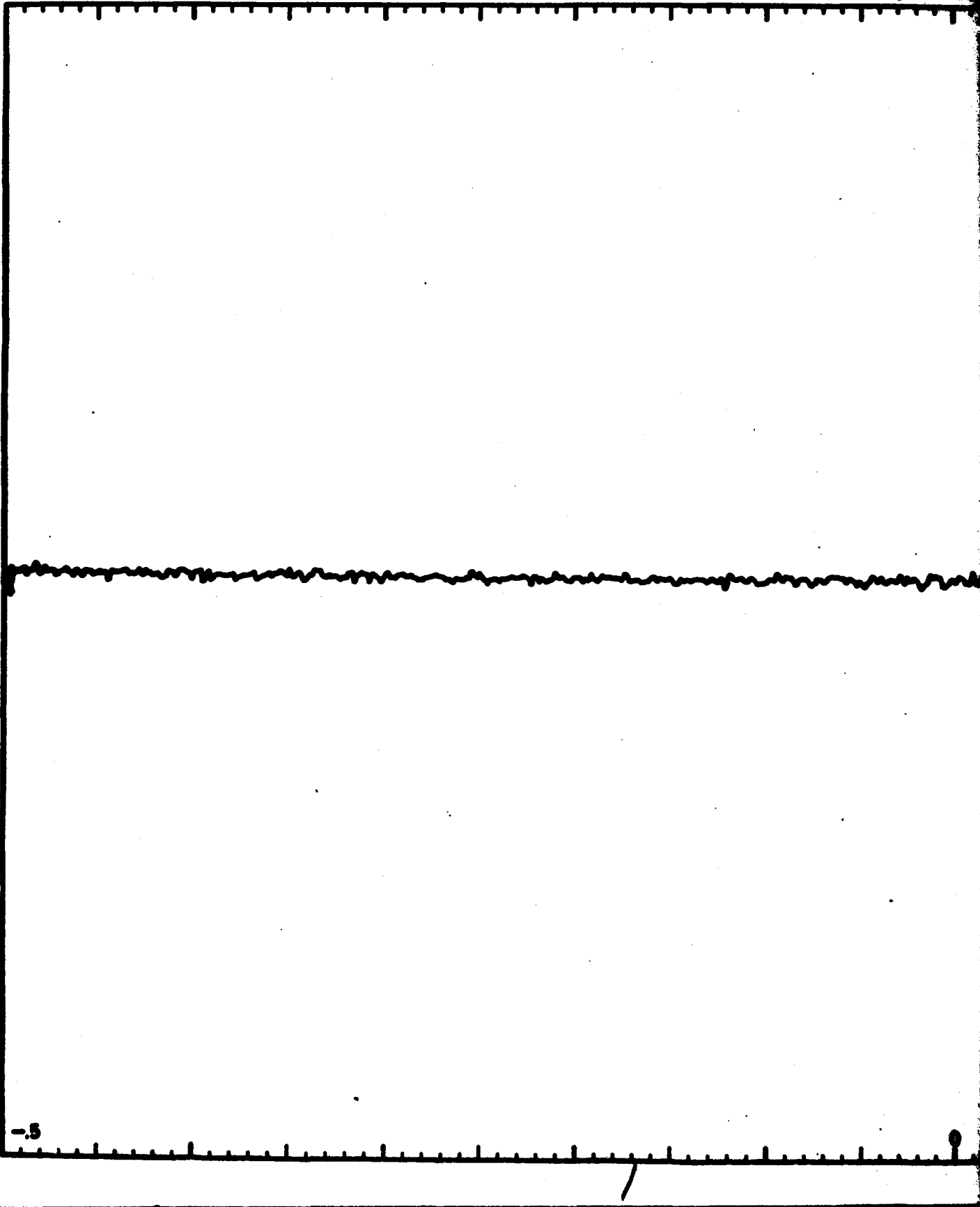
Spectrum No.

ESR-5

+5



4 x .00 0.250 2.5 x 1 6.3 x 10<sup>3</sup> 0.56  
Scan Range Time Constant Modulation Amplitude Receiver Gain Microwave Power  
3300 0 6 100 K AT 9.280  
Field Size Scan Time Modulation Frequency Temperature Microwave Frequency



J. Bennett

12/22/81

Remarks

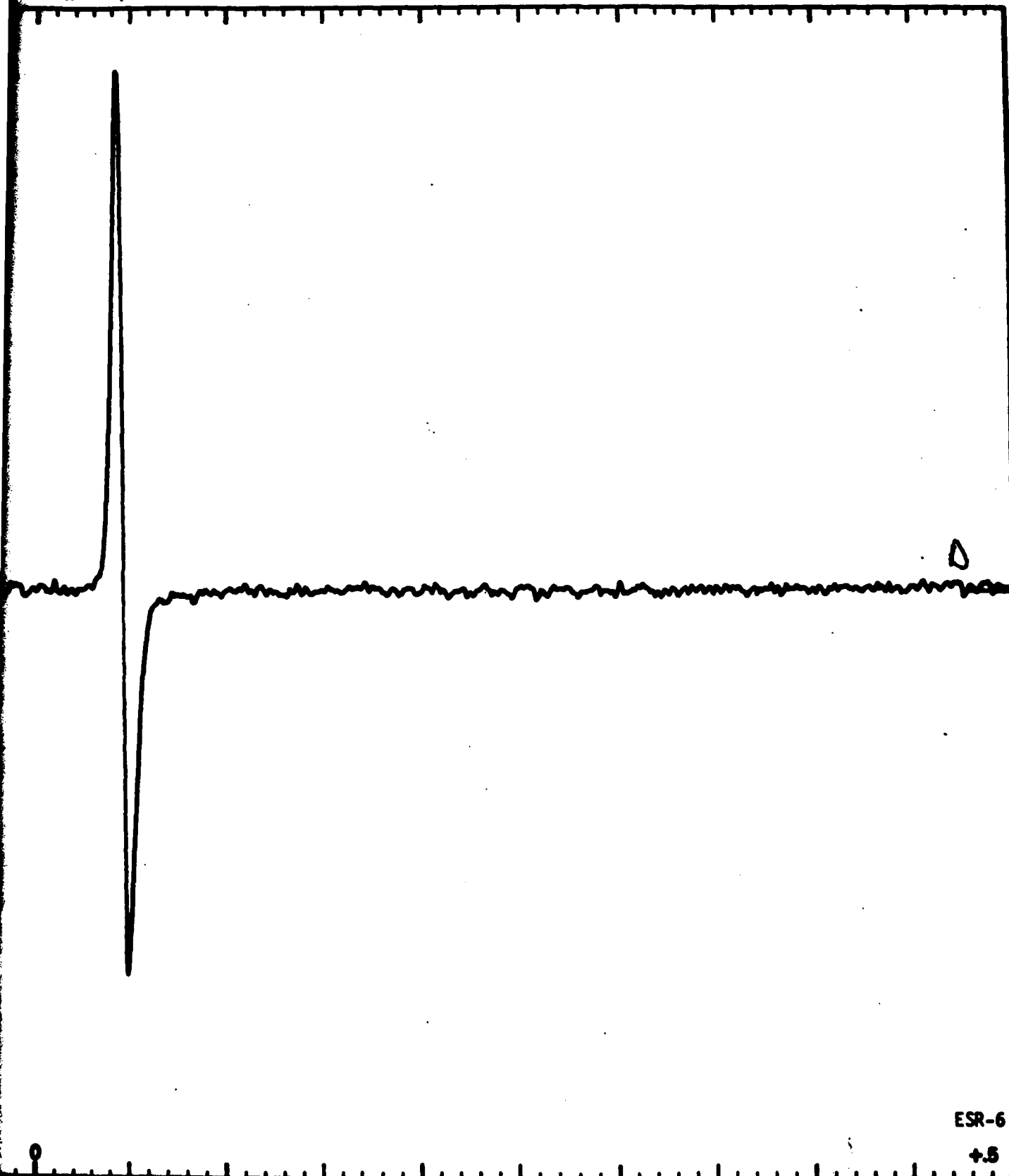
ESR CHART A

Sample 100% Anhydrous Ethanol <sup>old Bennett</sup>  
Date 10/10/81 ratonny mpanee

Spectrum No.

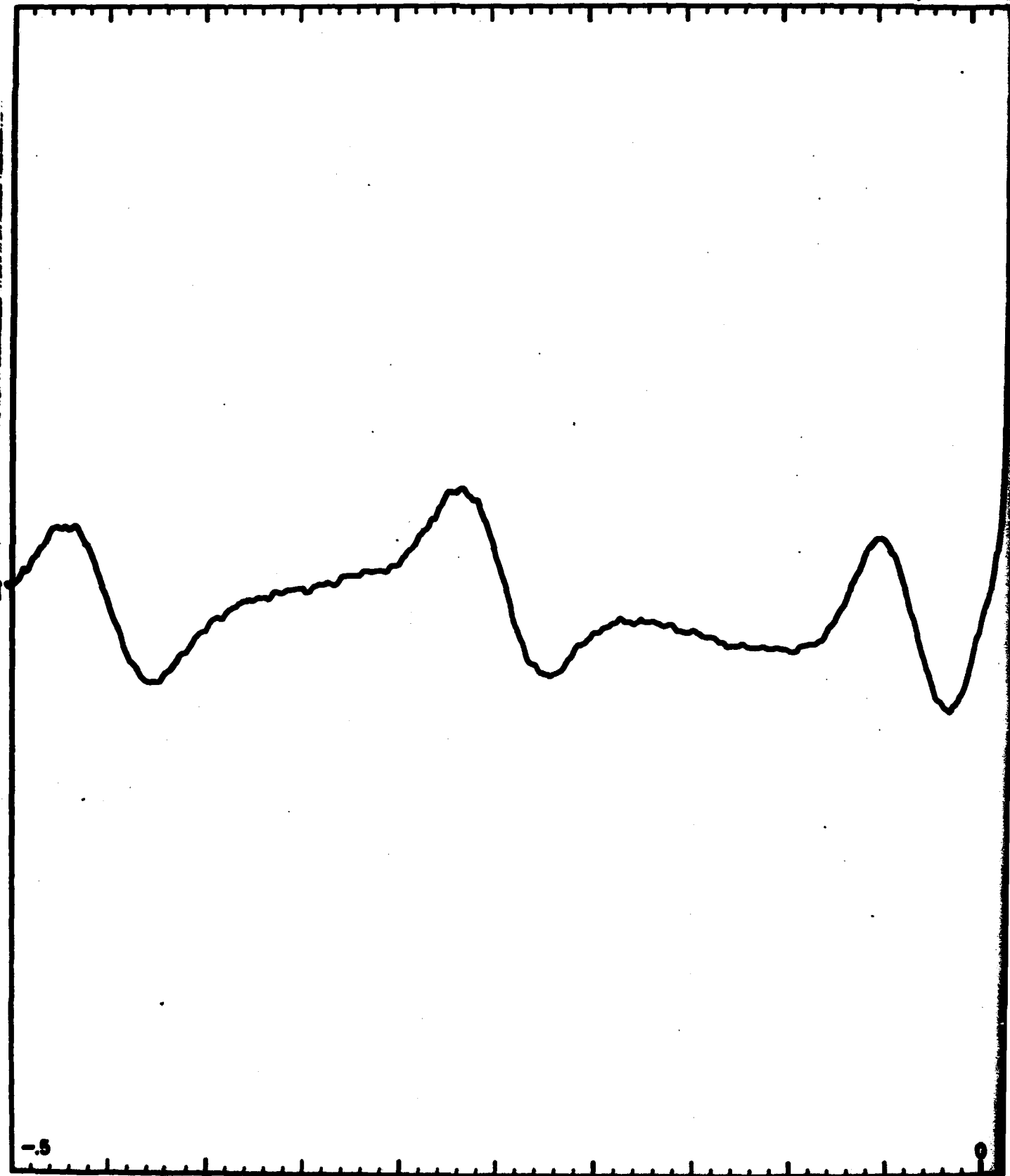
ESR-6

+5





Scan Range 4 x 100 g Time Constant 1.0 sec Modulation Amplitude 2.0 x 10 g Receiver Gain 10 x 10<sup>3</sup> Microwave Power 4.7 mW  
Field Set 3300 g Scan Time hrs 16 min Modulation Frequency 100 K Hz Temperature RT °C Microwave Frequency 9.283 GHz



Operator J. Bennett

Date 12/29/81 Remarks 5<sup>35</sup> pm

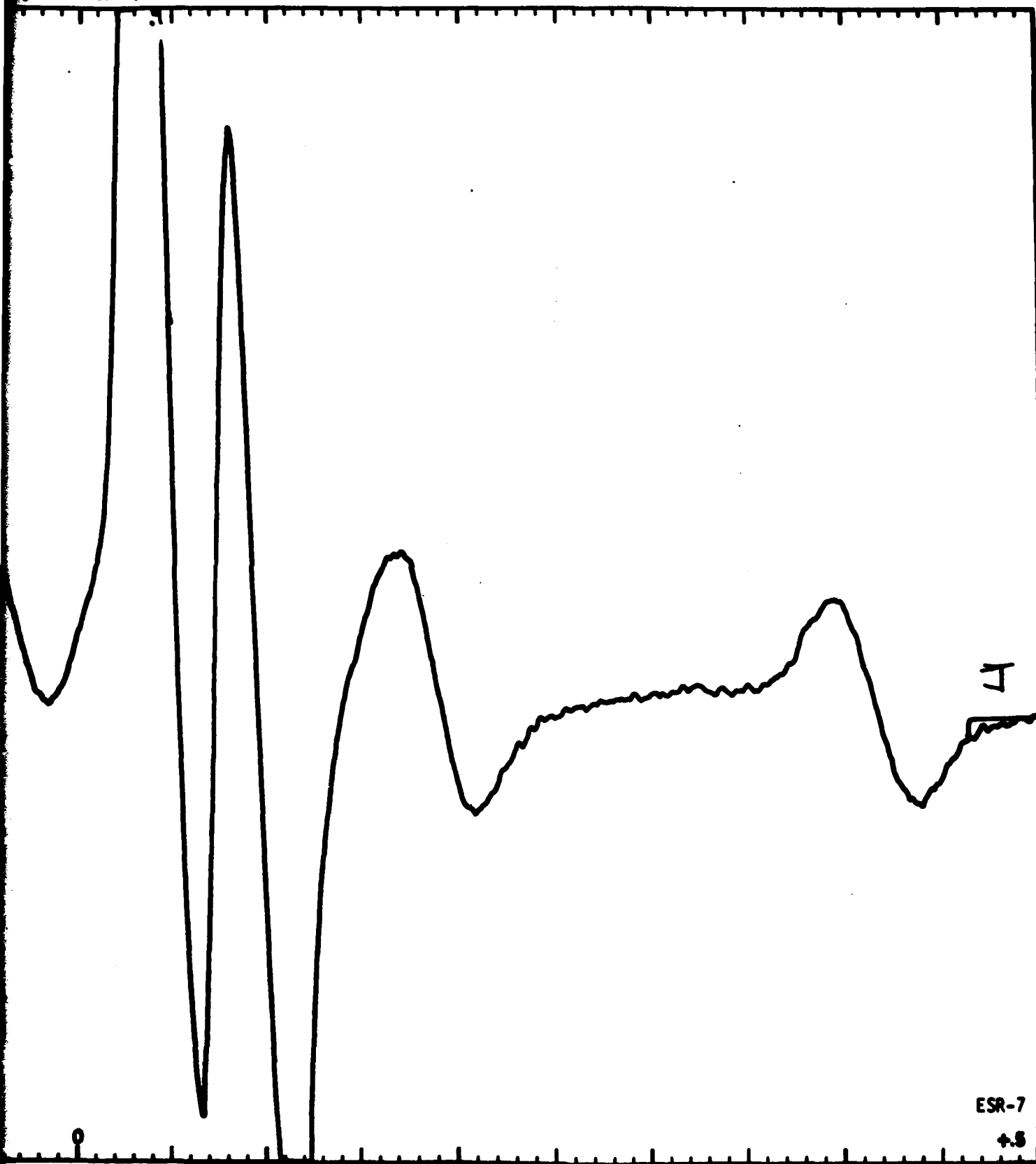
SPR CHART A

Sample 50% Oxidized Electrolyte 12/29/81 CT043

Spectrum No.

ESR-7

+5



% TRANSMITTANCE

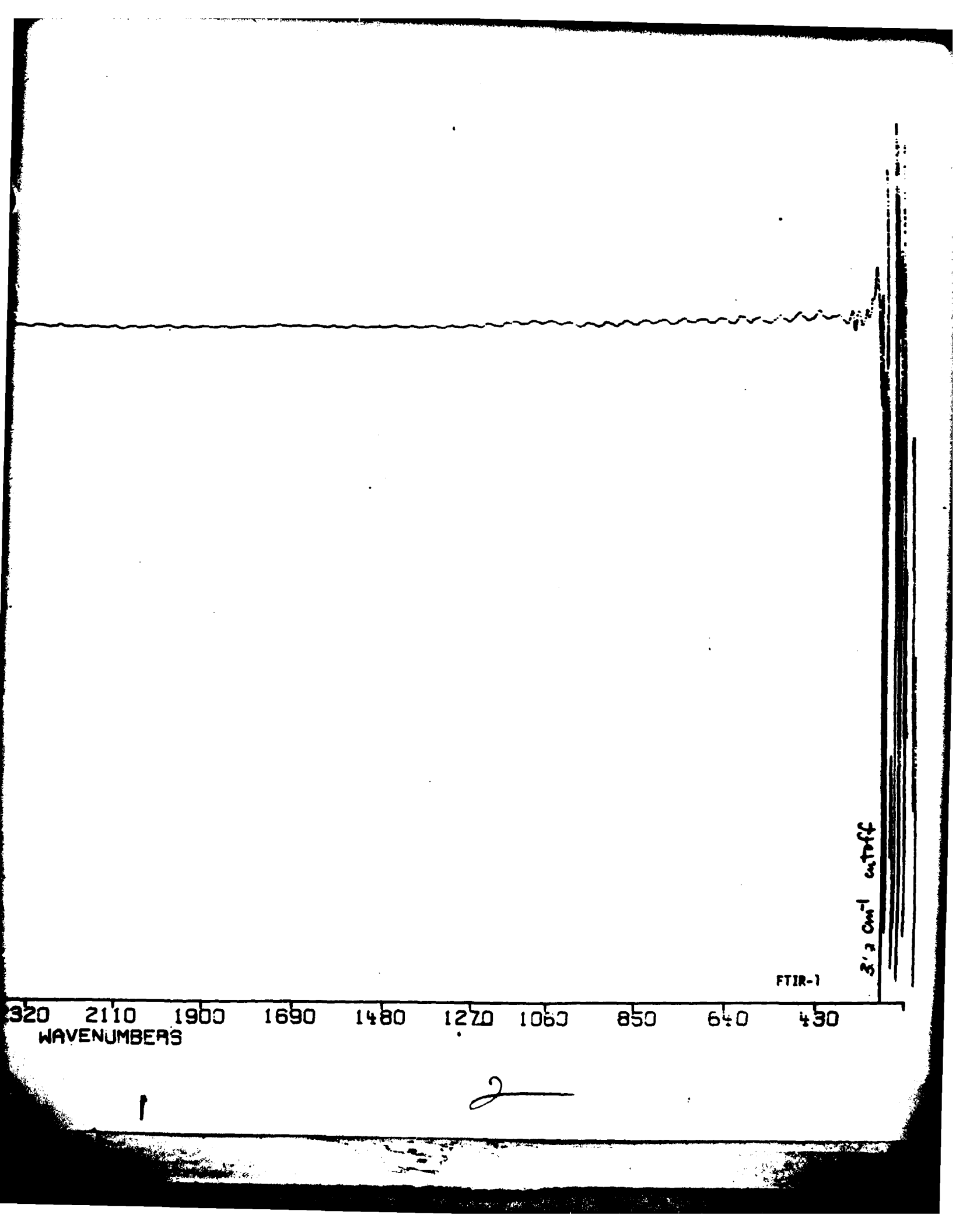
175.00 138.00 101.00 64.00 27.00 10.00 47.00 84.00 121.00 158.00

11/10/81

AGCL CELL BLANK  
*compensated*

000 3790 3580 3370 3160 250 2740 2530 2320

WAV



FTIR-1

3.2 cm<sup>-1</sup> cutoff

320 2110 1900 1690 1480 1270 1060 850 640 430  
WAVENUMBERS

1

2

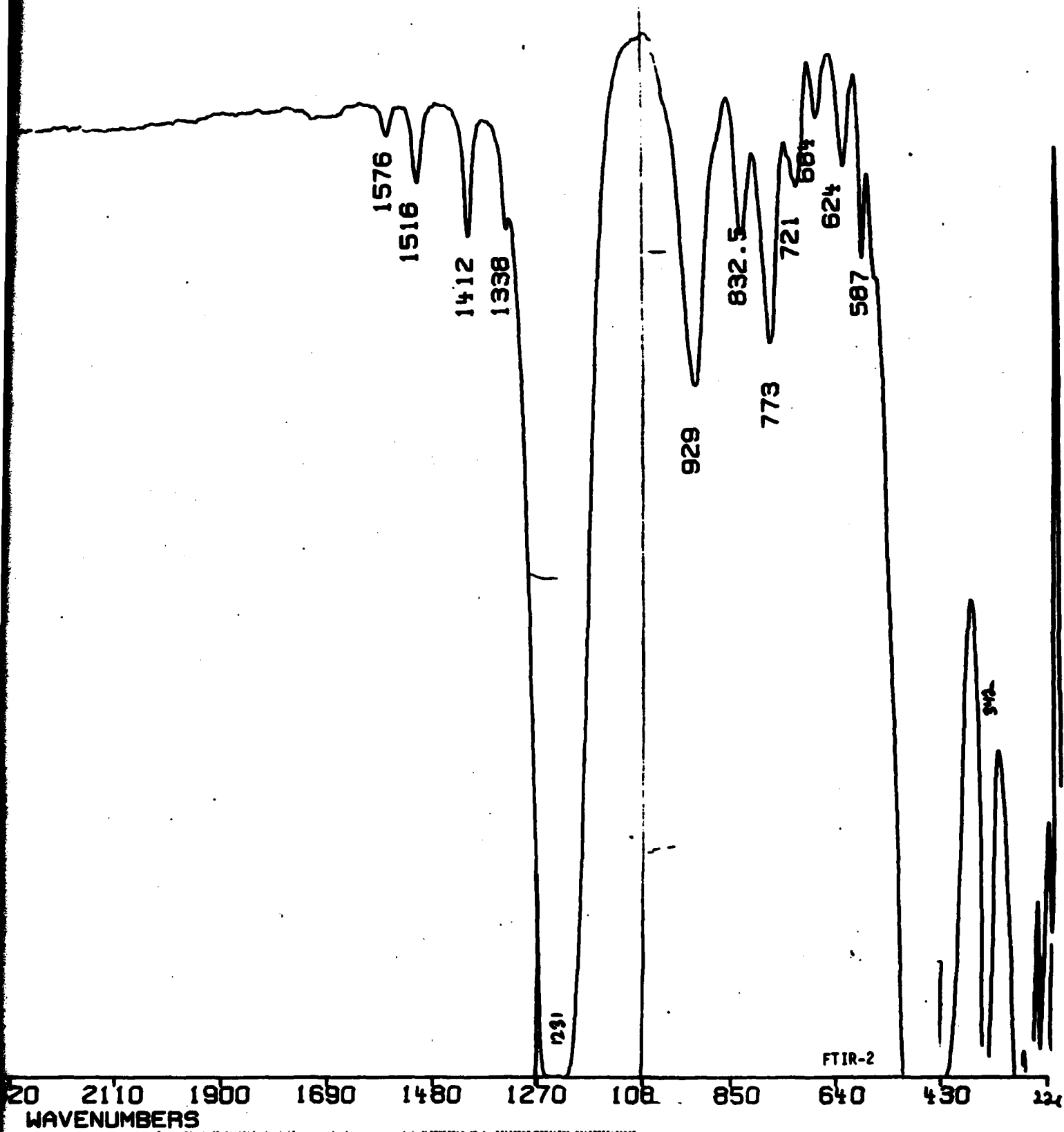
% TRANSMITTANCE

.00 6.00 12.00 18.00 24.00 30.00 36.00 42.00 48.00 54.00

DISTILLED SOCL2

2445.8

4000 3790 3580 3370 3160 2950 2740 2530 2320



1

2

POCL3

% TRANSMITTANCE

40.00 10.10 20.20 30.30 40.40 50.50 60.60 70.70 80.80 90.90

4000

3780

3580

3370

3160

2950

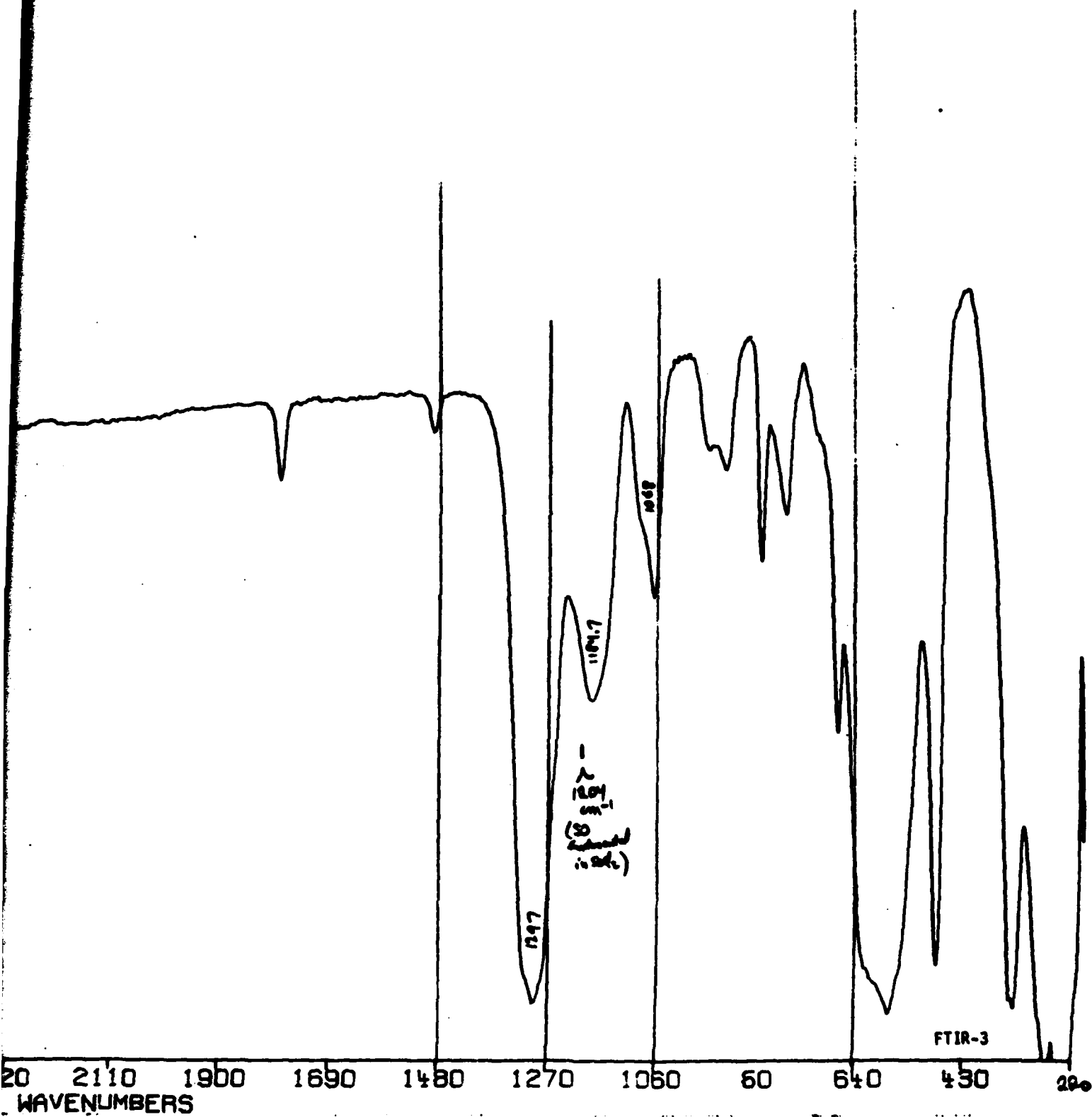
2740

2550

2320

2

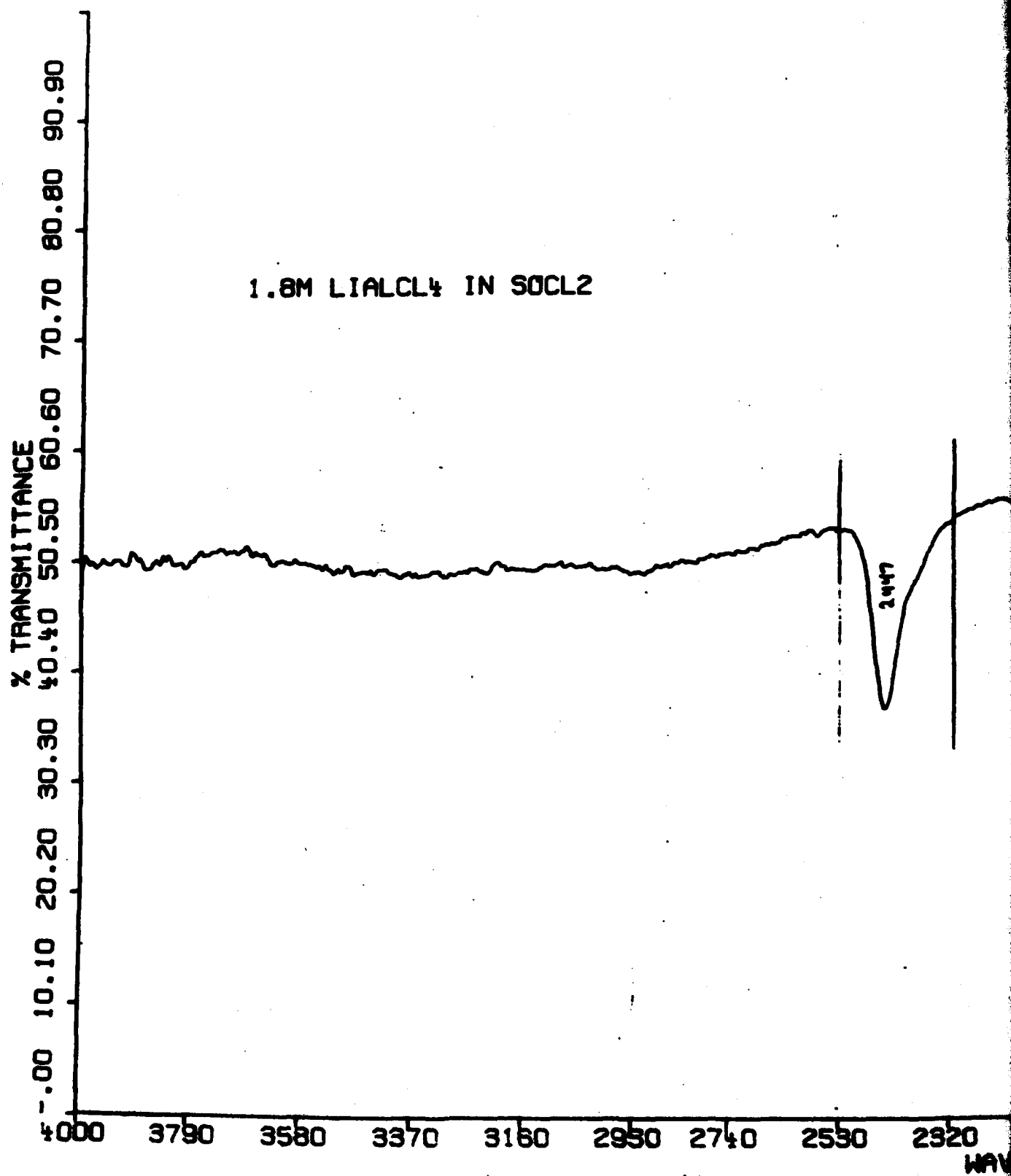
WAVES

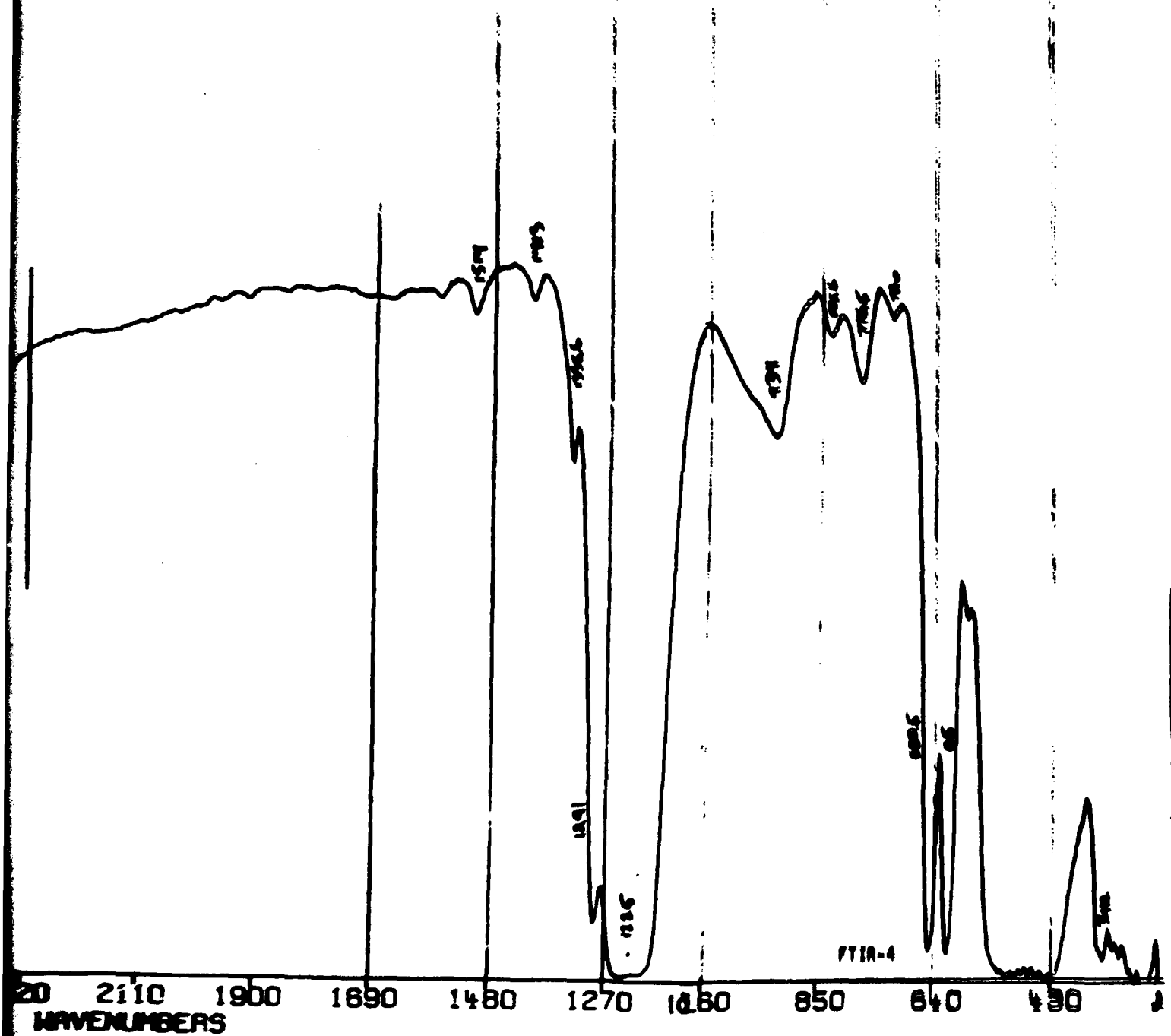


1

2







2

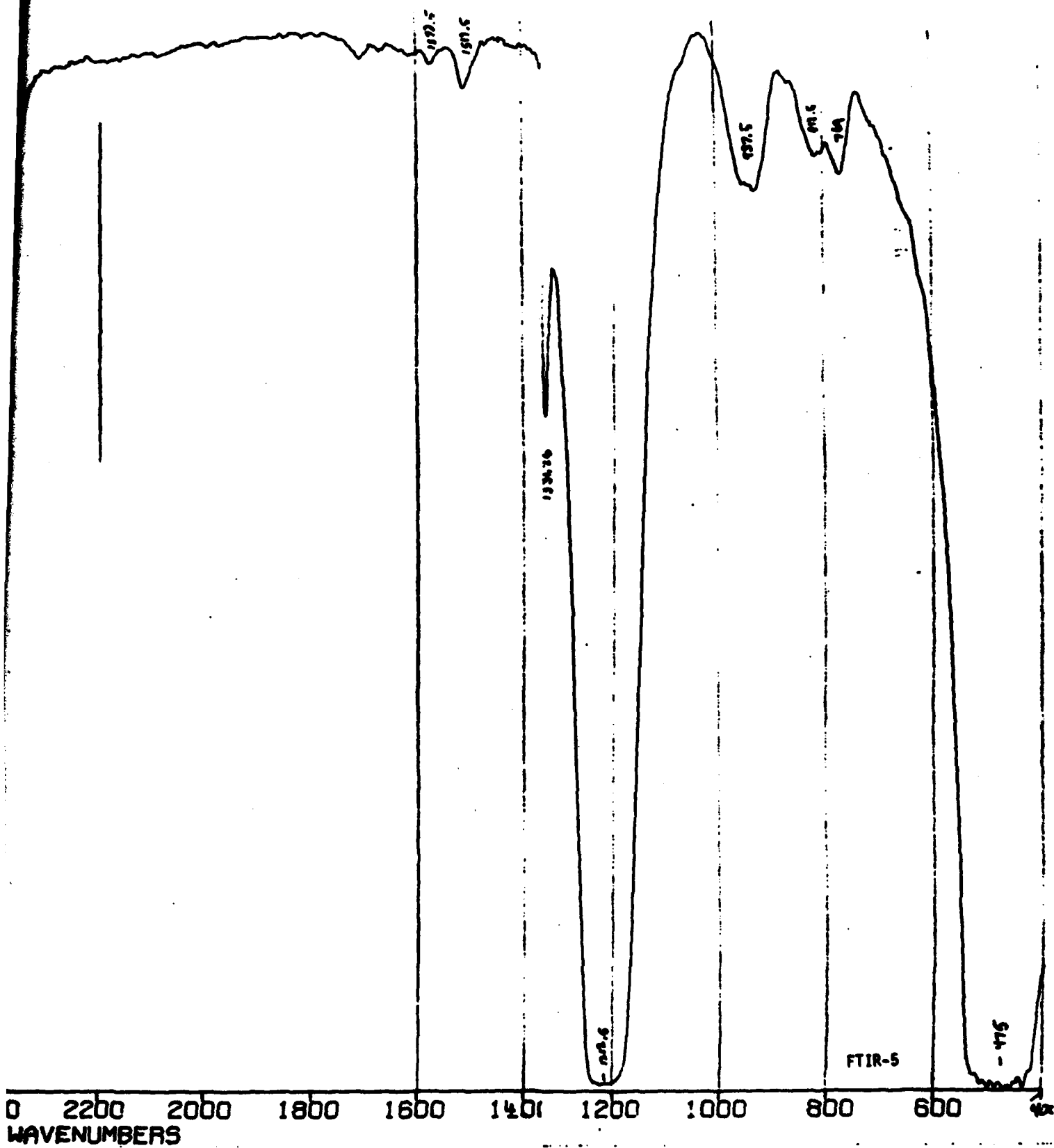
% TRANSMITTANCE

4.29 5.20 10.70 16.20 21.70 27.20 32.70 38.20 43.70 49.20

S IN ELECTROLYTE

4000 3800 3600 3400 3200 3000 2800 2600 2400 2200

WAVENUMBERS



1

2

% TRANSMITTANCE

4000 3800 3600 3400 3200 3000 2800 2600 2400

1 ATM SO<sub>2</sub> IN ELECTROLYTE

2455

WAV



FTIR-6

2200 2000 1800 1600 1400 1200 1000 800 600 400

WAVENUMBERS

1

2

40.00 10.10 20.20 30.30 40.40 50.50 60.60 70.70 80.80 90.90

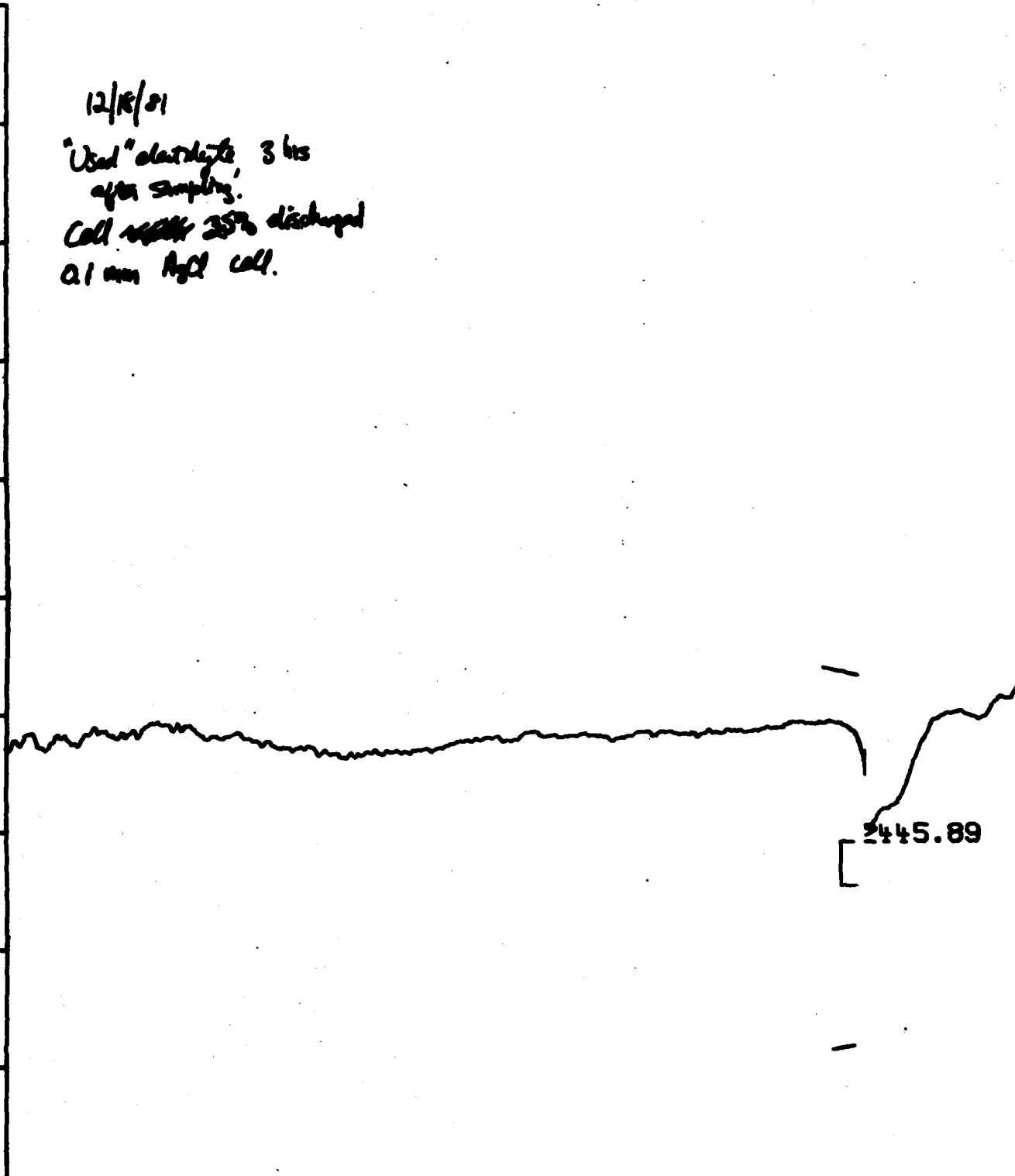
% TRANSMITTANCE

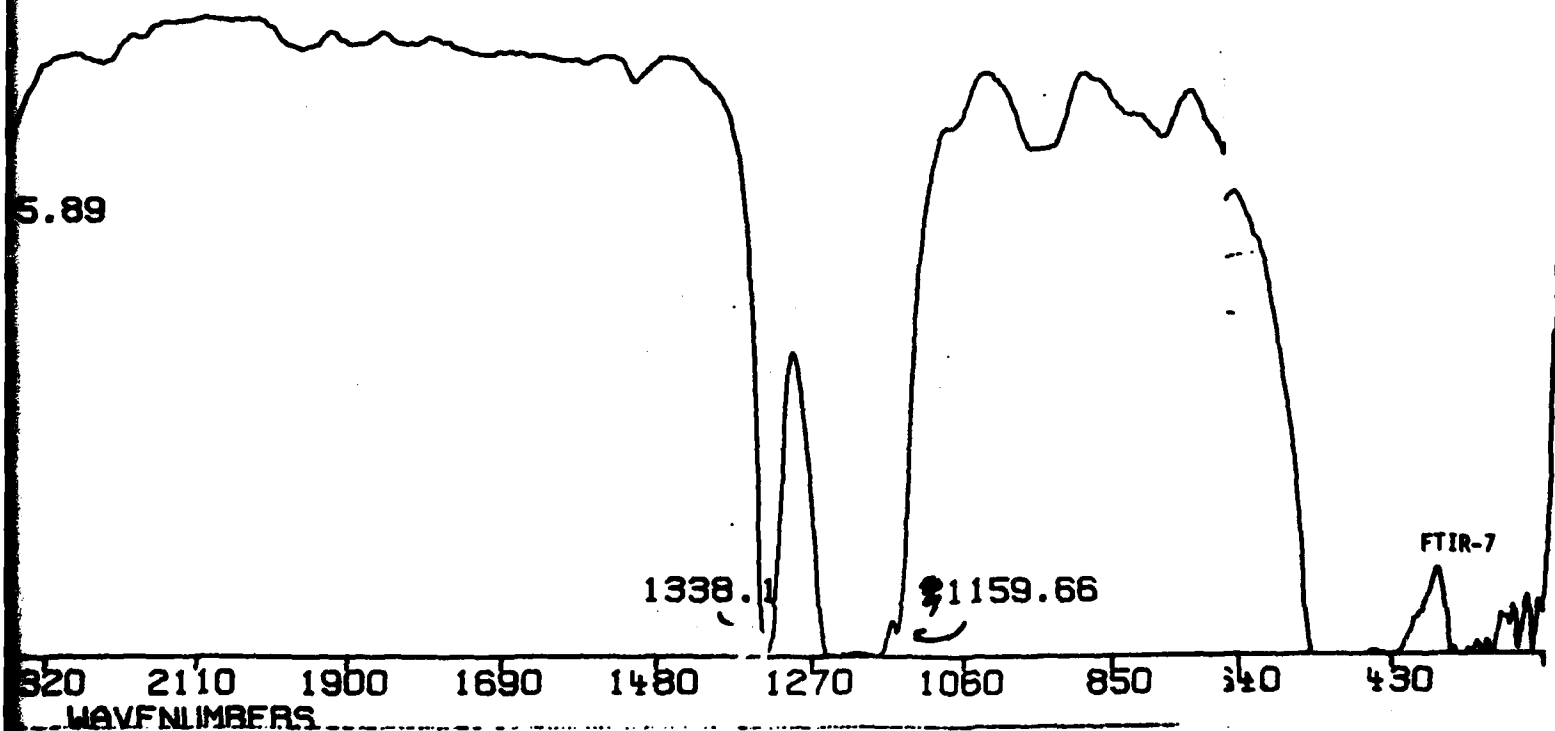
12/15/01  
"Used" standard, 3 hrs  
after sampling.  
Cell ~~with~~ 25% discharged  
0.1 mm AgCl cell.

4000 3790 3580 3370 3160 2950 2740 2530 2320

Wave

2445.89





1

2



SAMPLE: 25% DISCHARGED CELL 3 DAYS. FTER SAMPLING

SAMPLE: 25% DISCHARGED CELL 3 DAYS. FTER SAMPLING

XX

% TRANSMITTANCE

4.90 2.80 10.50 18.20 25.90 33.60 41.30 49.00 56.70 64.40

4000 3790 3580 3370 3160 2950 2750 2530 2320

2438

WAV

ING

958.8

1331

1152

FTIR-8

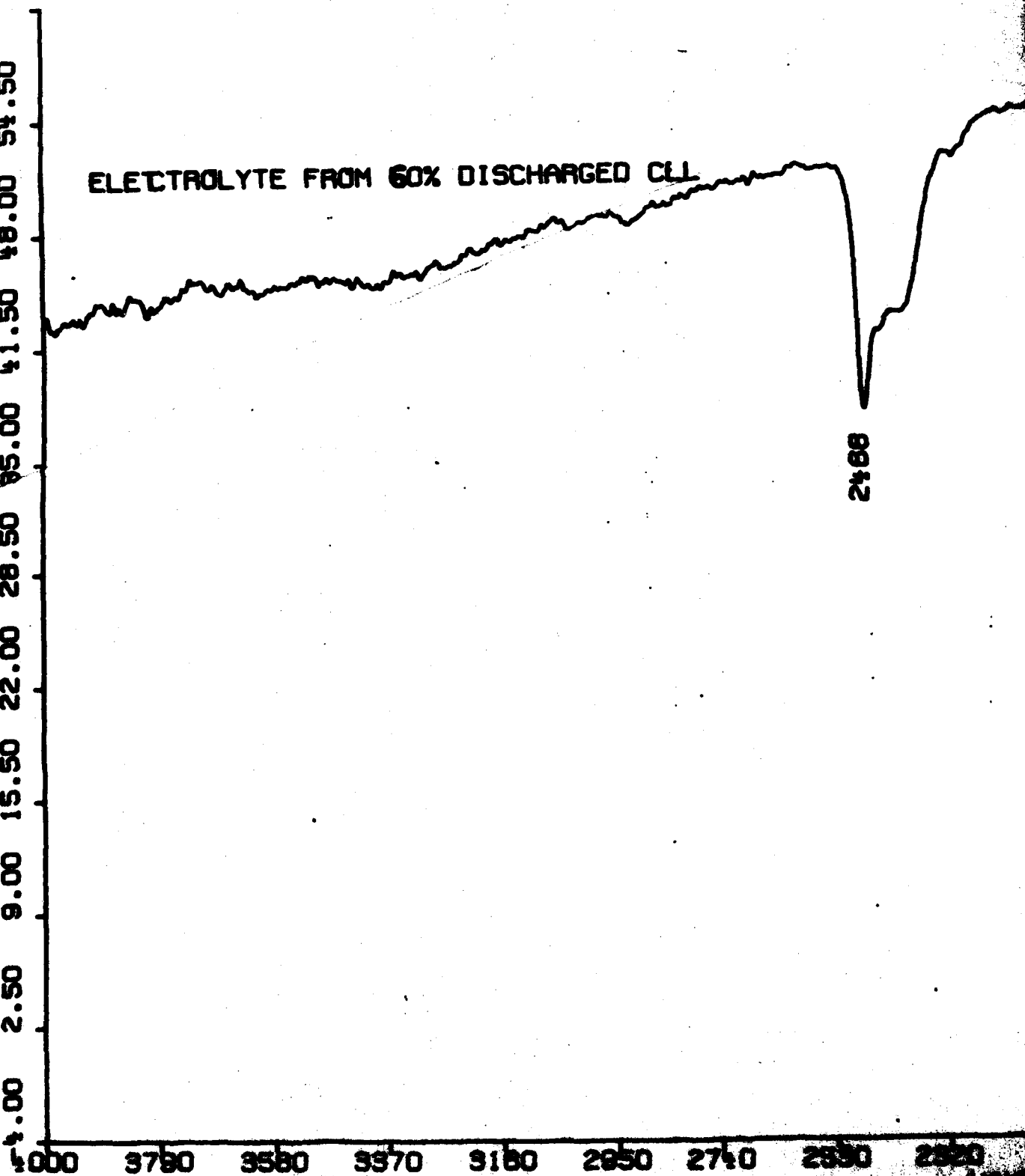
2320 2110 1900 1690 1480 1270 1060 850 640 430  
WAVENUMBERS

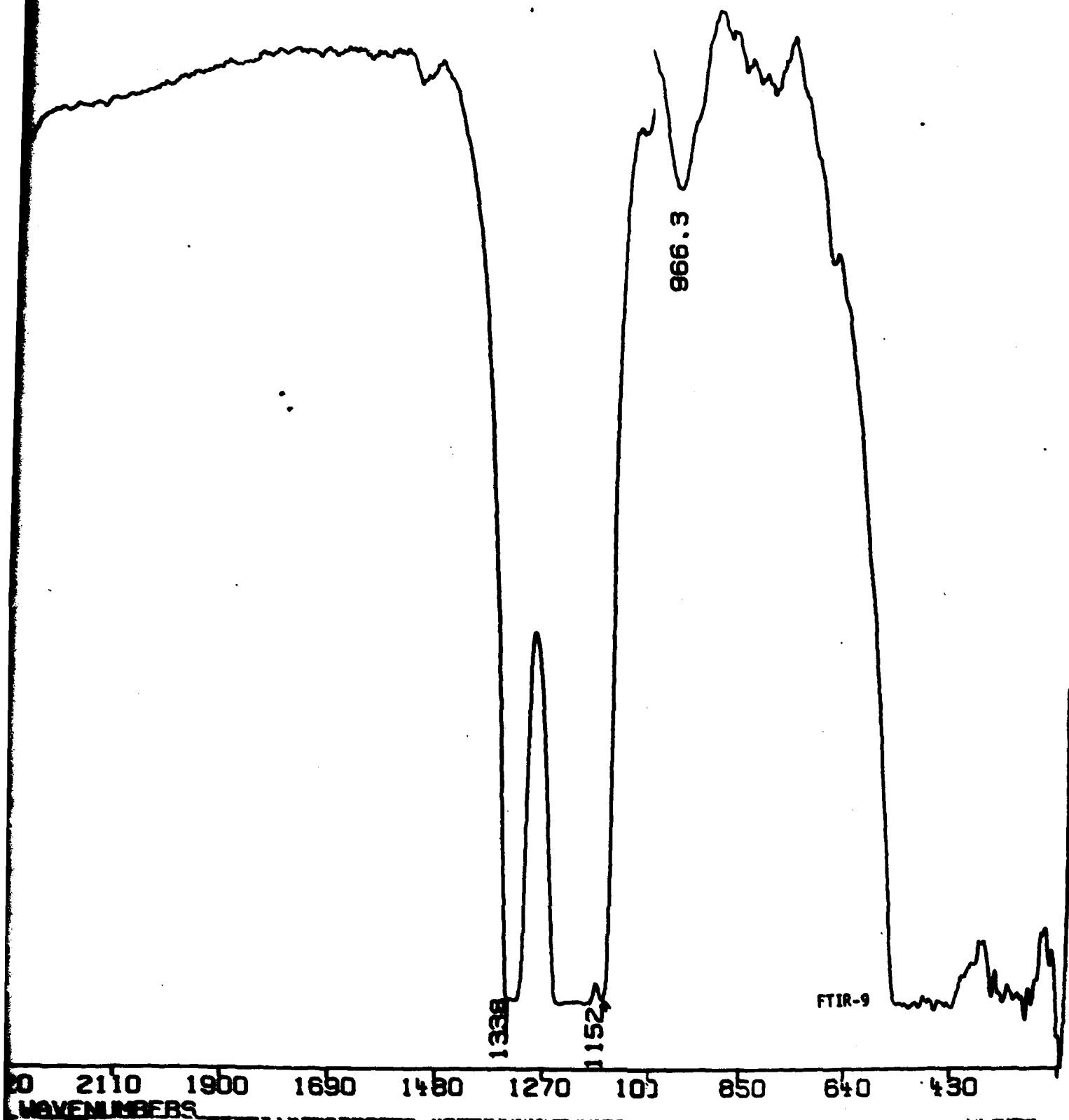
1

2

% TRANSMITTANCE

4.00 2.50 9.00 15.50 22.00 28.50 35.00 41.50 48.00 54.50





1

2

% TRANSMITTANCE

.19 5.60 11.00 16.40 21.80 27.20 32.60 38.00 43.40 48.80

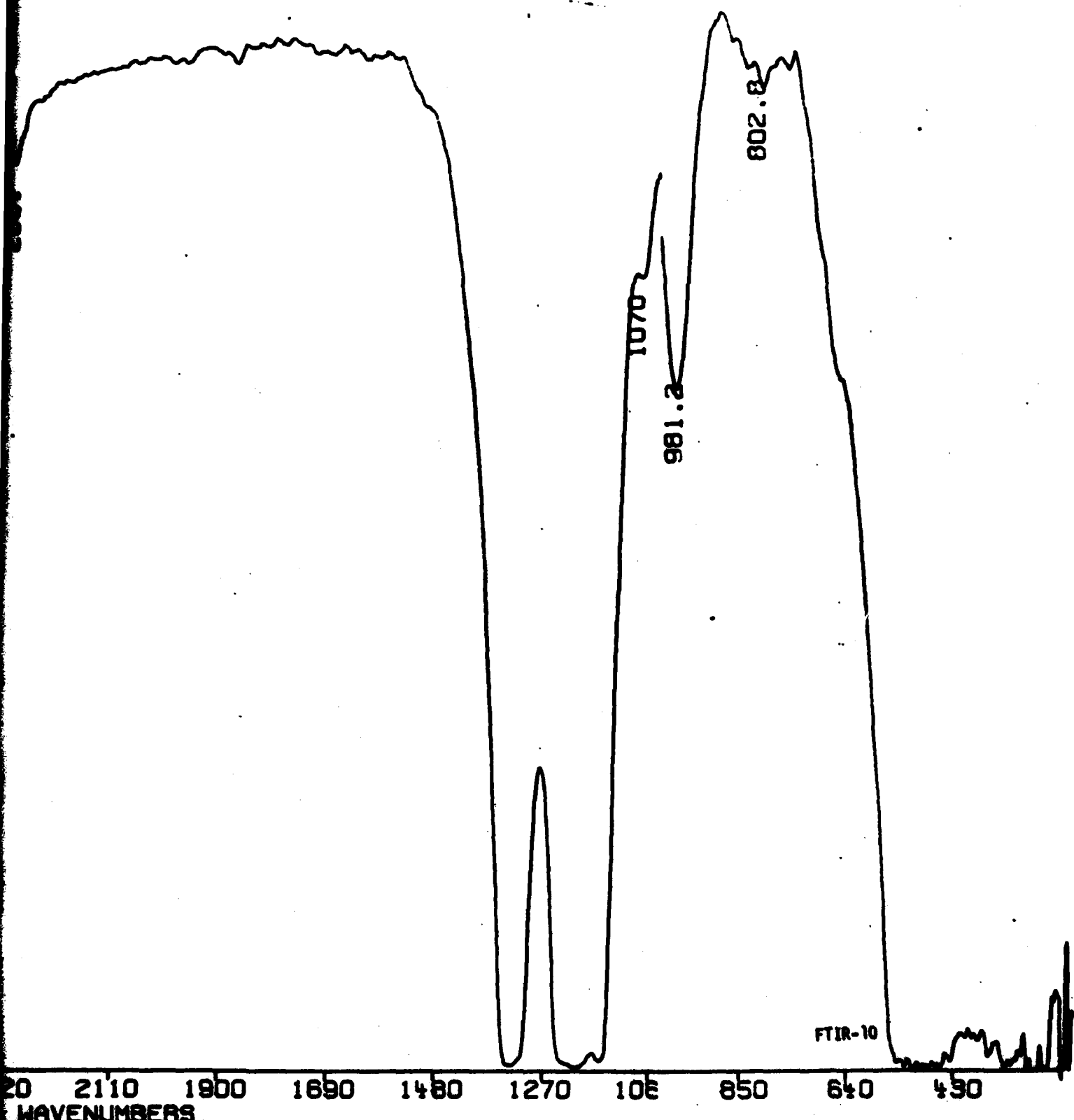
4000 3790 3580 3370 3160 2950 2740 2530 2320

POSITIVE ELECTROLYTE FROM 100% DISCHARGE CELL  
PDSP TONY.001

2475

1041

2304



1

2

SAMPLE: ELECTROLYTE FROM 100% DISCHARGED CELL  
(AFTER 7 DAYS STANDING)

% TRANSMITTANCE

57.80 50.40 43.00 35.60 28.20 20.80 13.40 6.00 1.40 0.80

4000 3750 3580 3370 3160 2950 2740 2530 2320

2300

